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Volume 4 DECEMBER, 1934 Number 3

Journal of Sedimentary Petrology

A Publication of the Society of Economic Paleontologists and Mineralogists
a Division of
The American Association of Petroleum Geologists

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NOTICE

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JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 4, NO. 3, PP. 103–110, TABLE I,
DECEMBER, 1934

PHYSICO-CHEMICAL FACTORS AFFECTING THE SOLUBILITY OF CALCIUM CARBONATE IN SEA WATER

ROGER REVELLE
Scripps Institution of Oceanography, La Jolla, California

ABSTRACT

Recent investigations of the relationships between the various forms of carbonic and boric acids in sea water have made it possible to delimit experimentally the solubility of calcium carbonate in the sea and to estimate quantitatively the relative effects on the solubility of variations in salinity, temperature, hydrostatic pressure, and carbon dioxide content. Although the experimental results obtained by various workers for the solubility product are not in agreement, it is at least certain that surface sea water at a temperature of 30° C is saturated with calcium carbonate. A table is presented showing the comparative effects on the solubility of changes in salinity, temperature, depth, and carbon dioxide content. It is shown that except for water in equilibrium with the atmosphere the most important factor controlling the solubility of $CaCO_3$ in sea water is the CO_2 content of the water, which, in turn, is chiefly dependent upon the nature and amount of biological activity. The order of importance of the other factors is temperature, salinity, and hydrostatic pressure. For water in equilibrium with the atmosphere, a condition probably only rarely attained, changes in temperature have the greatest effect on the solubility.

The solubility of calcium carbonate in sea water has been investigated by many workers, but until recently most of them have approached the problem only qualitatively or theoretically, although some have employed quantitative but usually indirect methods. It was pointed out by Johnston and Williamson (1916) in a paper which has become classic that the precipitation and solution of calcium carbonate in the sea is governed by the mass law equation, $[Ca^{++}] \times [CO_3^{=}] = K'_{CaCO_3}$, in which the brackets represent molar concentrations and K'_{CaCO_3} is the apparent or stoichiometric solubility product constant of calcium carbonate in sea water, that is, the limiting product of the concentration of calcium and carbonate ions in sea water of any given salinity and temperature in equilibrium with solid calcium carbonate. It may be seen from this equation that the three elements which control the solubility of $CaCO_3$ under any set of conditions are the concentrations of calcium and carbonate ions and the value of the constant K'_{CaCO_3}.[1] These factors are in turn dependent on salinity, temperature, hydrostatic pressure due to depth below the surface, carbon dioxide content, and the concen-

[1] This is true even though the concentration of carbonate ions is not a measure of the solubility of calcium carbonate, since most of the so-called dissolved calcium carbonate in any solution is present as calcium and bicarbonate rather than as calcium and carbonate ions.

tration of hydrogen and hydroxyl ions, as indicated by the pH.

The development of the necessary analytical methods and the application, by groups of workers in Europe and in this country, of the modern theories of strong electrolytes to the investigation of the problem have made it possible to delimit experimentally the three elements of the above equation, and to estimate quantitatively the relative effects on the solubility produced by variations in salinity, temperature, hydrostatic pressure, carbon dioxide content, and pH.

The total concentration of calcium may be measured directly, using the rapid and accurate micro-method recently developed by Kirk and Moberg (1933) or, as is done by Wattenberg (1933), the variation in the total calcium may be determined from the variations in the titratable base or alkalinity of sea water, provided the ratio between these two constituents is constant. The titratable base is defined as the equivalent concentration in milliequivalents per liter of strong bases balanced against weak acid radicals. In normal surface sea water at a temperature of 20° the calcium concentration and the titratable base generally bear constant ratios to the chlorinity, namely, $Ca/Cl = 0.02156$ and $B/Cl = 0.121$ to 0.125. For sea water of salinity 34 parts per thousand, the amount of calcium is about 400 milligrams per kilogram, equal to a molar concentration of approximately 10^{-2}.

On the other hand, the concentration of carbonate ions cannot be measured directly, but may be calculated by means of equations derived from the familiar mass law expressions:

$$\frac{\alpha_{H^+} \times [HCO_3^-]}{[H_2CO_3]} = K'_1, \text{ and}$$

$$\frac{\alpha_{H^+} \times [CO_3^=]}{[HCO_3^-]} = K'_2$$

in which α_{H^+} represents the activity or effective concentration of hydrogen ions, as indicated by the pH. The symbols K'_1, and K'_2 represent the apparent proportionality constants, respectively, of the first and second steps of the dissociation of carbonic acid in sea water, and the brackets, as before, indicate molar concentrations. The value of K'_1 in sea water having a salinity of 34 parts per thousand at a temperature of 20°C has been found to be close to 10^{-6} by a committee appointed by the International Council for the Exploration of the Sea to investigate the carbon dioxide equilibrium relationships of sea water (Buch, Harvey, Wattenberg, Gripenberg, 1932). According to Buch (1930, 1933a, b), K'_2, under the same conditions, is close to 10^{-9}, and this value has also been obtained in experiments at the Scripps Institution (Moberg, Greenberg, Revelle, Allen, 1934). It was found (Revelle and Moberg, 1934) that boric acid, which is present to the extent of about 18 per cent of the titratable base and which has an apparent dissociation constant in sea water close to K'_2 of carbonic acid, plays an ap-

preciable role in the buffer mechanism. A correction for boron must be applied to the total titratable base in order to determine the amount of the base which is balanced against carbonic acid. At pH 8.2, this correction, according to Buch (1933a, b) is equal to 4 per cent of the titratable base, or 30 per cent of the content of carbonate ions. If this value be accepted as correct, the amount of carbonate expressed as CO_2 at pH 8.20 in sea water of salinity 34 $^0/_{00}$ and temperature 20°C will be about 11 mg per liter, equal to a molar concentration of 2.5×10^{-4}.

The first and second apparent dissociation constants of carbonic acid in sea water vary with the salinity of the water according to the relations:

$$pK'_1 = 6.47 - .154\sqrt[3]{\text{salinity } ^0/_{00}}$$

$$pK'_2 = 10.45 - .368\sqrt[3]{\text{salinity } ^0/_{00}} - .0063 \text{ salinity } ^0/_{00}$$

in which pK'_1 and pK'_2 represent the logarithms of the reciprocals of K'_1 and K'_2. The temperature coefficient of pK'_1 at 20° is -0.006 per degree, at 5° it is -0.009. At 20° the value of $\Delta pK'_2/\Delta T$ is -0.011, at 5° it is -0.012. Carbonic acid thus becomes an apparently stronger acid with both increasing salinity and temperature. Buch and Gripenberg (1932) have presented evidence to show that pK'_2 and pK'_1 decrease 0.05 and 0.02 units, respectively, for each 1000 meters of depth. That this change in the dissociation constants has little effect on the solubility of calcium carbonate is shown in table 1, part b.

It may be seen from the analytical figures for calcium and carbonate, given above, that the average ion product for calcium carbonate in surface sea water of salinity 34 $^0/_{00}$ at 20° and pH 8.20 is 2.5×10^{-6}. Revelle and Fleming (1934), following the technique employed by Gee and Moberg (1932), raised the pH of numerous samples of sea water at 30° by removing carbon dioxide, and succeeded in precipitating calcium carbonate in the form of aragonite needles and spherulites. Precipitation continued to take place at least until an average ion product for calcium and carbonate of 2.37×10^{-6} was obtained. From these results they concluded that the solubility product constant of calcium carbonate at 30° is about 2.4×10^{-6}, and that surface sea water under tropical conditions of temperature is approximately saturated with calcium carbonate. On the other hand, Wattenberg (1933), from the results of experiments in which calcite was dissolved in waters the pH of which had been lowered through addition, at 20° and 20 parts per thousand chlorinity, of carbon dioxide, concluded that K'_{CaCO_3} of surface water is 1.0×10^{-6} and that, consequently, surface water is greatly oversaturated. Wattenberg later revised his calculations to take account of the boron content of sea water, and his value for K'_{CaCO_3} is correspondingly lowered to 0.6×10^{-6}.

Part of the discrepancy between the two sets of values for K'_{CaCO_3} is doubtless due to the facts that the results of Revelle and Fleming were obtained on aragonite, which is

possibly more soluble in sea water than calcite (although it is aragonite which is precipitated in such regions as the Bahama Banks), and that the grain sizes of the particles precipitated by them were probably smaller, and hence more soluble, than those of the particles dissolved by Wattenberg. On the other hand, if the value given by him for the solubility product constant, together with his values for its variation with temperature and salinity, are correct, sea water of salinity 36 °/₀₀ and temperature 5°C is saturated with calcium carbonate at pH values above 7.66. According to Wattenberg, there are very few places in the South Atlantic where the pH, when corrected for temperature, falls below 7.70. It is difficult to reconcile the presence of red clay, supposedly the residue remaining after the solution of calcareous materials, with the supersaturated condition of the water which would exist if Wattenberg's value for K'_{CaCO_3} is correct. Although it is probable that neither our results nor those of Wattenberg represent true equilibrium conditions, which are very difficult to attain, they at least indicate the limits of the value of the solubility product constant.

Regardless of the absolute value of K'_{CaCO_3} its relative changes with temperature and over a small range of salinity are known with fair certainty. Between 34 and 36 °/₀₀ salinity, according to Wattenberg's data, the change for each part per thousand of salinity in pK'_{CaCO_3}, the logarithm of the reciprocal of K'_{CaCO_3}, is -0.038. Wattenberg has also calculated that $\frac{\Delta pK'_{CaCO_3}}{\Delta T}$ is $+0.010$ per degree at 20° and $+0.005$ at 5°. The effect of depth (hydrostatic pressure) on K'_{CaCO_3} is unknown, but Johnston and Williamson (1916) believed that it is small.

The knowledge of the rates of change of the solubility product constant with respect to changes in its controlling factors, together with the data previously given for the other components of the system, enable us to calculate the relative effects on the solubility of calcium carbonate of variations in salinity, temperature, depth, and carbon dioxide content of the atmosphere and of the water. At saturation,

$$\frac{[Ca^{++}] \times [CO_3^{=}]}{K'_{CaCO_3}} = 1.$$

In a supersaturated solution this quotient is greater than one and in an undersaturated solution it is less than one, by amounts which are proportional to the degree of supersaturation or undersaturation of the solution. Changes in the relative value of the quotient, regardless of its absolute value, may thus be taken to represent changes in the degree of saturation.

Table 1 shows the comparative effects of changes in salinity, temperature, depth, and carbon dioxide content on the components of the system controlling the solubility of calcium carbonate and on the relative value of the quotient mentioned above. Two sets of conditions are postulated.

A. SURFACE WATER. CARBON DIOXIDE CONTENT IN EQUILIBRIUM WITH THAT OF ATMOSPHERE

Postulated Original Conditions:—Salinity, 34‰; Temperature, 20°C; P_{CO_2}, 3.50×10^{-4} Atmospheres

Factors of the System	c_s *mM* / liter	$K'_1 \times 10^9$	$K'_2 \times 10^9$	B *mEq* / liter	B_C *mEq* / liter	pH	$\sum CO_2$ *mM* / liter	H_2CO_3 *mM* / liter	HCO_3^- *mM* / liter	$CO_3^=$ *mM* / liter	Ca^{++} *mM* / liter	$K'_{CaCO_3} \times 10^6$	$\frac{[Ca^{++}] \times [CO_3^=]}{K'_{CaCO_3}}$
Values for postulated conditions.	34.2	1.07	.90	2.32	2.23	8.14	2.02	.0120	1.78	.223	10.4	2.32[a]	1.00
Salinity raised to 36‰. Temperature, P_{CO_2} unchanged	33.9 −1%	1.10 +3%	.97 +8%	2.45 +6%	2.36 +6%	8.15 +.01 unit	2.11 +5%	.0119 −1%	1.85 +4%	.253 +13%	11.0 +6%	2.53 +9%	1.10 +10%
Temperature raised to 25°C. Salinity, P_{CO_2} unchanged	29.8 −13%	1.15 +7%	1.02 +13%	2.32 —	2.22 −.5%	8.16 +.02 unit	1.98 −2%	.0104 −13%	1.71 −4%	.252 +13%	10.4 —	2.06 −11%	1.26 +26%
P_{CO_2} lowered to 2.50×10^{-4} atmospheres. Salinity, temperature unchanged.	34.2 —	1.07 —	.90 —	2.32 —	2.21 −1%	8.26 +.12 unit	1.95 −4%	.0085 −29%	1.66 −7%	.273 +22%	10.4 —	2.32 —	1.22 +22%
pH raised 0.1 unit. ($P_{CO_2} = 2.63 \times 10^{-4}$). Salinity, temperature unchanged.	34.2 —	1.07 —	.90 —	2.32 —	2.21 −1%	8.24 +.10 unit	1.96 −3%	.0090 −25%	1.69 −5%	.264 +18%	10.4 —	2.32 —	1.18 +18%

[a]Assumed value.

B. DEEP WATER.

Postulated Original Conditions:—Salinity, 36‰; Temperature, 5°C; depth, 1000 m; total CO_2, 2.35 mM/liter.

Factors of the System	$K'_1 \times 10^6$	$K'_2 \times 10^9$	B *mEq* / liter	B_{CO_2} *mEq* / liter	pH	$\sum CO_2$ *mM* / liter	H_2CO_3 *mM* / liter	$HCO_3^=$ *mM* / liter	$CO_3^=$ *mM* / liter	Ca^{++} *mM* / liter	$K'_{CaCO_3} \times 10^6$	$\frac{[Ca^{++}] \times [CO_3^=]}{K'_{CaCO_3}}$
Values for postulated conditions.	.95	.69	2.47	2.43	7.85	2.35	.033	2.21	.108	11.1	1.20[b]	1.00
Salinity lowered to 34‰ Temperature, depth, and base/total CO_2 ratio unchanged.	.92 −3%	.64 −7%	2.33 −6%	2.29 −6%	7.88 +.03 unit	2.22 −6%	.030 −9%	2.09 −6%	.102 −6%	10.4 −6%	1.10 −8%	.97 −3%
Temperature lowered to 0°C. Salinity, depth, and base/total CO_2 ratio unchanged.	.85 −10%	.60 −14%	2.47 —	2.43 —	7.91 +.06 unit	2.35 —	.032 −3%	2.21 —	.107 −1%	11.1 —	1.25 +5%	.95 −5%
Depth increased to 5000 m. Salinity, temperature, and base/total CO_2 ratio unchanged.	1.47 +36%	.82 +18%	2.51 +2%	2.47 +2%	7.75 −.10 unit	2.39 +2%	.027 −18%	2.26 +2%	.105 −3%	11.3 +2%	1.20 —	.99 −1%
Total CO_2 increased to 2.40 mM/liter. Salinity, temperature, depth, and titratable base unchanged.	.95 —	.69 —	2.47 —	2.44 +.5%	7.72 −.13 unit	2.40 +2%	.046 +39%	2.27 +3%	.083 −23%	11.1 —	1.20 —	.77 −23%
pH lowered 0.1 unit (total CO_2=2.39 mM/liter). Salinity, temperature, depth, and titratable base unchanged.	.95 —	.69 —	2.47 —	2.44 +.5%	7.75 −.10 unit	2.39 +2%	.042 +27%	2.26 +2%	.088 −19%	11.1 —	1.20 —	.81 −19%

[b]Assumed value.

The first is that of water at the sea surface in equilibrium with atmospheric carbon dioxide having a salinity of 34 ‰, temperature of 20°, and carbon dioxide partial pressure of 3.50×10^{-4} atmospheres. For simplicity, this water is assumed to be at the saturation point, in other words, K'_{CaCO_3} is assumed to equal 2.32×10^{-6}. The table shows the relative effects on the state of the system and on the relative solubility of calcium carbonate of changes of salinity from 34 to 36 ‰ of temperature from 20° to 25°, of CO_2 pressure from 3.5 to 2.5, and of pH from 8.14 to 8.24. The marked increase in the degree of saturation due to a five-degree rise in the temperature (owing chiefly to changes in the solubility of carbon dioxide) is very striking, as is the large effect of relatively small changes in carbon dioxide content, as measured by the pH.

The second set of postulated conditions is that of water at an initial depth of 1000 meters, not in equilibrium with the atmosphere, having an original salinity of 36 ‰, temperature of 5°, and total carbon dioxide content of 2.35 millimoles per liter. In order to simplify the calculations, the water is again assumed to be at the saturation point, in other words, K'_{CaCO_3} is assumed to equal 1.20×10^{-6}. This part of the table shows the relative effects on the state of the system and on the relative solubility of calcium carbonate, of changes in salinity from 36 to 34 ‰, of temperature from 5 to 0°, of total CO_2 from 2.35 to 2.40, and of depth from 1000 to 5000 meters.

From a study of the table we may conclude that, except for water in equilibrium with the atmosphere, a condition probably only rarely established, the most important factor controlling the solubility of $CaCO_3$ in sea water is the CO_2 content of the water, which is chiefly dependent upon the nature and amount of biological activity. The order of importance of the other physical and chemical factors considered in this report is temperature, salinity, and hydrostatic pressure.

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EXPLANATION OF SYMBOLS AND CALCULATIONS USED IN MAKING THE TABLE

P_{CO_2} = partial pressure of carbon dioxide, as atmospheres.

c_s = solubility of carbon dioxide in salt solution, as millimoles per liter under a partial pressure of one atmosphere of carbon dioxide (values of c_s from Bohr (1899) after Buch and others (1932).

K'_1 = first apparent dissociation constant of carbonic acid in sea water $= \dfrac{[\alpha_{H^+}] \times [HCO_3^-]}{[H_2CO_3]}$.

According to Buch and others, (1932)

$$pK'_1 = \log \frac{1}{K_1'} = 6.47 - .154\sqrt[3]{\text{salinity } ‰} \text{ at } 20° \text{ C.}$$

$$\frac{\Delta pK'_1}{\Delta T} = -0.006 \text{ (at } 20°) = -0.009 \text{ at } 5°.$$

$$\frac{\Delta pK'_1}{\Delta \text{depth}} = -0.048 \text{ per 1000 meters.}$$

K'_2 = second apparent dissociation constant of carbonic acid in sea water $= \dfrac{\alpha_{H^+} \times [CO_3^-]}{[HCO_3^-]}$.

According to Moberg and others (1934),

$$pK'_2 = 10.45 - 0.368\sqrt[3]{\text{salinity } ‰} - 0.0063 \text{ salinity } ‰.$$

According to Buch and others (1932),

$$\frac{\Delta pK'_2}{\Delta T} = -0.011 \text{ (at } 20°) = -0.012 \text{ at } 5°.$$

$$\frac{\Delta pK'_2}{\Delta \text{depth}} = -0.018 \text{ per 1000 meters.}$$

B = titratable base = the equivalent concentration, expressed as milliequivalents per liter, of strong bases balanced against weak acid radicals in sea water = Salinity ‰ × 0.0682 (at 20°) (Wattenberg, 1933).

B_{CO_3} = titratable base balanced against carbonate and bicarbonate = $B - [H_2BO_3^-]$, where

$[H_2BO_3^-] = \frac{K'_{H_2BO_3} \times [H_2BO_3]}{\alpha_{H^+} + K'_{H_2BO_3}}$. Values of $K_{H_2BO_3}$ from Buch (1933b). $[H_2BO_3]$ (as milliequivalents at 20°) = salinity °/₀₀ × .0125, (after Moberg and Harding (1933)).

$pH = \log \frac{1}{\alpha_{H^+}}$, where α_{H^+} equals the effective molar concentration of hydrogen ions in sea water.

$$(\alpha_{H^+})^2 - \frac{[H_2CO_3]}{B_{CO_2}} \times K'_1 \alpha_{H^+} - 2 \frac{[H_2CO_3}{B_{CO_2}} \times K'_1 K'_2 = 0.$$

also,

$$(\alpha_{H^+})^2 + K'_1 \left(\frac{\sum CO_2}{B_{CO_2}} - 1 \right) \alpha_{H^+} - K'_1 K'_2 \left(2 \times \frac{\sum CO_2}{B_{CO_2}} - 1 \right) = 0.$$

$\sum CO_2$ = total carbon dioxide = the sum of the free carbon dioxide, bicarbonate, and carbonate ions present in sea water, as millimoles per liter = $\frac{B_{CO_2}}{1 + \frac{2K'_2}{\alpha_{H^+}}} \times \left(\frac{\alpha_{H^+}}{K'_1} + \frac{K'_2}{\alpha_{H^+}} + 1 \right)$

H_2CO_3 = free carbon dioxide, as millimoles per liter = $P_{CO_2} \times c_s$ (Henry's law)

$$= \frac{B_{CO_2}}{1 + \frac{2K'_2}{\alpha_{H^+}}} \times \frac{\alpha_{H^+}}{K'_1}$$

HCO_3^- = bicarbonate ion concentration, as millimoles per liter = $\frac{B_{CO_2}}{1 + \frac{2K'_2}{\alpha_{H^+}}}$

$CO_3^=$ = carbonate concentration, as millimoles per liter = $\frac{B_{CO_2}}{1 + \frac{2K'_2}{\alpha_{H^+}}} \times \frac{K'_2}{\alpha_{H^+}}$

Ca^{++} = calcium ion concentration, as millimoles per liter = salinity °/₀₀ × 0.3052 (after Thompson and Wright, 1930; Kirk and Moberg, 1933; and Wattenberg, 1933.)

K'_{CaCO_3} = apparent solubility product constant of calcium carbonate in sea water, that is, the limiting product of the concentration of calcium and carbonate ions in sea water of any given salinity and temperature in equilibrium with solid calcium carbonate. In a solution saturated with calcium carbonate,

$$\frac{[Ca^{++}] \times [CO_3^=]}{K'_{CaCO_3}} = 1.$$

In a supersaturated solution this ratio is greater than 1; in an undersaturated solution the ratio is less than 1.

According to Wattenberg (1933)

$$\frac{\Delta pK'_{CaCO_3}}{\Delta T} = +0.010 \text{ (at } 20°) = +0.005 \text{ (at } 5°)$$

$$\frac{\Delta pK'_{CaCO_3}}{\Delta \text{ salinity}} = -0.038 \text{ (between 34 and 36 °/₀₀)}$$

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PRELIMINARY REMARKS ON THE CALCAREOUS SHALLOW-WATER MARINE DEPOSITS OF FLORIDA AND THE BAHAMAS

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ABSTRACT

A forthcoming publication will give the results of a four-year study of the shallow-water marine deposits found along the coasts of southeastern Florida and the Bahamas. Calcareous organic skeletons and their broken parts were found to comprise about 96 per cent of the total deposit. The remaining 4 per cent consist of quartz, magnetite, mica, volcanic glass, and a few other mineral species. Chemically precipitated aragonite is discussed, as are calcareous oolites and ellipsoidal pellets, considered as of faecal origin. One hundred and fifty mechanical analyses are interpreted in relation to sorting and distributing agencies.

The calcareous shallow-water marine deposits of southern Florida and the Bahamas have attracted the attention of students of marine sediments for the past eighty years. Recently, a critical study of 74 representative samples from these regions, collected by T. W. Vaughan, was completed, and an abstract of the findings is here presented.

It was established that the deposits are predominantly composed of the skeletal remains of organisms. At least 73 per cent of the material was definitely identified as organic, and most of the silt and clay, totalling about 23 per cent, is thought to have a similar origin. Only about 4 per cent, representing minerals, oolites, and a few aggregates is classified as inorganic.

The quantitative values of the four major constituents in the 74 samples are: (1) calcareous algae, 22.8 per cent; (2) mollusk shells, 15.8 per cent; (3) foraminiferal tests, 11.7 per cent; and (4) Madreporarian corals, 9.0 per cent. Excluding silt and clay, which averaged 23.4 per cent, the combined quantity of all other constituents is 17.3 per cent.

Non-calcareous minerals are present in small quantities, except a part of the northern Florida reef tract, where quartz is relatively abundant. Their source is thought to be the Pleistocene formations of the mainland and particularly the Miami oolite.

The small but widespread amount of volcanic glass and a few minerals such as olivine, magnetite, mica, and some quartz are interpreted as having been wind-blown. Coal and ashes, brought from outside sources by human agencies are found incorpo-

rated in the sediments of Tortugas lagoon and, in smaller amounts, in a few other places.

Ellipsoidal pellets, found abundantly along and near the west end of South Bight, Andros Island, are interpreted as being of corpolitic origin. It is suggested that this explanation may not apply to all pellets of similar form found in marine sediments.

Calcareous oolites are quantitatively evaluated and discussed in connection with their distribution and their possible formation by physico-chemical processes.

Chemically precipitated aragonite needles were recognized in the finest portions of almost every sample, but their production is thought to be limited to a few small areas and that currents transport them to other areas. In this connection a review is given of recent studies of the buffer mechanism of sea water and of its bearing on the solubility and precipitation of calcium carbonate in the ocean.

Mechanical analyses of the 74 carefully studied samples and of 76 samples previously described by others were plotted as histograms in order to study the distribution of particle sizes. In Tortugas lagoon the smaller particles are accumulating in the troughs while the coarser material is left on the tops and slopes of the ridges. This distribution corresponds to that observed by Trask in the Channel Island region off the California coast. The shallow lee-side flat off the west end of South Bight, Andros Island, Bahamas, is covered by fine-grained muds and oozes because there are no strong currents to carry away the fine particles. The upper and middle parts of the Florida reef-tract keys are subjected to variable currents which generally remove the fine material from exposed bottom sediments and cause varying degrees of sorting in the larger particles.

Sediments from the Florida and Bahama regions are compared with similar shallow-water calcareous deposits of the Great Barrier Reef, Australia; Samoa; Funa-futi Atoll; Maiao Island, South Pacific; and two calcareous beaches of high northern latitudes. The absence of aragonite needles in all other areas except Maiao Island indicates that conditions favorable to the precipitation of calcium carbonate from sea water are of rare occurrence.

JOURNAL OF SEDIMENTARY PETROLOGY, VOL. 4, NO. 3, PP. 113–126, FIGS. 1–3, TABLES I–VIII, DECEMBER, 1934

ON THE ROLE OF MICROORGANISMS IN THE PRECIPITATION OF CALCIUM CARBONATE IN THE DEPOSITS OF FRESH WATER LAKES[1]

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ABSTRACT

It has been found that many aerobic organisms isolated from the deposits of Lake Mendota as well as many commonly known bacterial species are capable of precipitating calcium carbonate under artificial conditions. Some evidence is presented that certain of them will precipitate the carbonate from solutions carrying the same amount of calcium as does lake water. Chemical and X-ray analyses have proven these crystals to be calcite which is the form of carbonate found in the deep muds of Lake Mendota. The results of two experiments, however, have failed to prove the connection of the activity of microorganisms and the precipitation of calcium carbonate within the deposits of Lake Mendota.

The problem of calcium carbonate precipitation by microorganisms is of interest geologically as a possible explanation of the origin of certain lime deposits. Several workers have investigated this problem, especially with reference to deposits in tropical seas.

It has been known since the work of Murray and Irvine (1889–90) that calcium carbonate may be precipitated from a calcium-bearing medium during protein decomposition. Several later investigators (Drew, 1911, 1914; Molisch, 1925; Kalantarian and Petrossian, 1932; Brussoff, 1933) have isolated organisms which will form calcium carbonate crystals in agar media containing soluble calcium salts. These workers have considered that the organisms isolated play an important role in calcium carbonate precipitation in nature. Naeslund (1927) has shown, however, that several commonly occurring aerobic organisms will precipitate calcium from solid media. It will be shown in the following that carbonate precipitation cannot be attributed to a definite species of organisms but to a large group which embraces many different species.

Lipman (1924, 1926, 1929) has criticized the claims of bacterial precipitation of lime on the basis of a limited number of experiments from which it appeared that bacteria cannot precipitate calcium in media made up from sea water and no ad-

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ditional calcium. Molisch (1925), however, has demonstrated that at least certain microorganisms will form carbonate in sea water media. With added calcium a marked increase in the precipitation could be noted. Lipman has stated that the peptone used by Molisch in his "sea water" medium was probably an additional source of calcium.

Bavendamm (1932) has given a complete review of the literature on calcium carbonate precipitation in sea waters.

It appears that the concentration of calcium may be a controlling factor for precipitation of calcium carbonate under conditions found in nature. Experiments along this line have been carried out and are described in this paper. The work was done with various microorganisms including microorganisms from the deposits in Lake Mendota, a fresh water lake in southern Wisconsin. The deposit of this lake consists of a black sludge which is rich in organic matter. This changes rather abruptly with depth to a grey mud or marl which is chiefly calcite. The amount of soluble calcium in the water of the deposits is from three to four milligrams per 100 cubic centimeters. The calcium content of the fresh water lakes of Wisconsin ranges from 0.05 to 5.0 milligrams per 100 cubic centimeters; this is considerably less than that of the open sea, which is approximately 45 milligrams per 100 cubic centimeters.

Several mechanisms have been suggested for the biological precipitation of calcium carbonate among which are:

1. $(NH_4)_2CO_3 + CaSO_4 \rightarrow (NH_4)_2SO_4 + CaCO_3$

(Murray and Irvine—1889–90).

The carbon dioxide is presumably produced during the respiration of the microorganisms and the ammonia from protein decomposition or nitrate reduction. It has been considered that this equation is the most likely as explaining the biological precipitation of calcium carbonate in sea waters.

2. Calcium lactate $+ 6O_2 \rightarrow 5H_2O + 5CO_2 + CaCO_3$

(Drew—1914).

This equation is probably of only limited importance due to the lack of an abundant supply of the organic salts of calcium in nature.

3. $CaSO_4$ + bacterial sulphur reduction $\rightarrow CaS$

$CaS + H_2O + CO_2 \rightarrow CaCO_3 + H_2S$

(Nadson—1928).

Nadson arrived at this equation by noting that hydrogen sulphide was produced in fermenting muds in conjunction with small white crystals within the material. No quantitative work was presented, however, and his conclusions are not well supported by data.

4. $Ca(HCO_3)_2 + 2NH_4OH \rightarrow (NH_4)_2CO_3 + 2H_2O + CaCO_3$

(Kellerman and Smith—1914).

Kellerman and Smith approached this problem from a physical-chemical standpoint and their mechanism

may therefore more nearly represent conditions of carbonate precipitation as found in nature.

Media to provide for the mechanisms proposed by Murray and Irvine and Drew were used to demonstrate carbonate precipitation under artificial conditions. These are presented in Table I. For solid media fifteen grams of agar were added to the above. Precipitation of the carbonate can be observed directly by examination of the colonies under the microscope. Crystals of characteristic shape are observed which will effervesce with dilute hydrochloric acid (Brussoff, 1933). This method has been used by previous investigators.

ORGANISMS CAPABLE OF PRECIPITATING CALCIUM CARBONATE FROM CALCIUM-BEARING AGAR MEDIA

New species of various organisms have been described by several recent investigators to which have been assigned the peculiar property of precipitating calcium carbonate. Molisch (1925) and Naeslund (1927), however, have shown that several common organisms will cause precipitation of the carbonate on suitable media. In order to test the ability of a number of bacterial species to precipitate carbonate on solid media, the following experiments were carried out.

Aerobic mud dilution plates.—Dilutions of mud from the bottom of Lake Mendota were made and plated on calcium sulphate and calcium lactate agar media. After four weeks of aerobic incubation the percentage of colonies showing precipitation was seen. In each case at least 100 colonies were observed. The results are given in Table II. It will be noticed that the percentage of colonies showing precipitation is in general somewhat higher in the muds from the

TABLE I. *Media used for the demonstration of calcium carbonate precipitation by certain microorganisms.*

Ingredients	Media				
	Calcium Sulphate	Calcium Lactate (1)	Molisch's (2)	Mud Extract	Modified Speakman's(3)
	grams	grams	grams	grams	grams
Peptone	5	1	10	—	5
Glucose	—	—	—	—	10
Glycerol	—	—	5	—	—
Ca lactate	—	5	—	—	—
$CaSO_4$	1	—	—	—	—
$CaCl_2$	—	—	—	—	varies
KNO_3	1	2	—	—	—
K_2HPO_4	—	—	—	—	0.5
KH_2PO_4	—	—	—	—	0.5
Na_2HPO_4	—	0.25	—	—	—
NaCl	—	—	—	—	0.01
$MnSO_4$	—	—	—	—	0.01
$FeSO_4$	—	—	—	—	0.01
Lake water	1000	1000	1000	—	—
Distilled water	—	—	—	—	1000
Mud extract	—	—	—	1000	—

(1) Lipman, 1924. (2) Molisch, 1925. (3) Speakman, 1923.

surface of the deposit than from the deeper layers. From the results obtained it is evident that the number of colonies on each plate that will form crystals varies between wide limits.

Anaerobic mud dilution plates.—Mud dilution plates were also poured with calcium sulphate, calcium lactate, Molisch's, modified Speakman's and 5 per cent Bacto-beef agar. The last three mentioned contained one-half of one per cent calcium chloride. The plates were incubated under anaerobic conditions at 37°C for three and one-half weeks. There was no evidence of precipitation on any of the colonies examined on these plates even though good growth did occur.

Algae.—Colonies of algae developing on dilution plates of media con-

TABLE II. *Percentage of colonies precipitating crystals after four weeks on calcium sulphate and calcium lactate agar media from mud dilution inoculations at two temperatures of incubation*

Sample number	Origin of sample	Calcium sulphate medium		Calcium lactate medium	
		10°C per cent	28°C per cent	10°C per cent	28°C per cent
26	Surface	—	7	—	47
27	Surface	—	20	—	36
28	Surface	—	8	—	31
29	Deep	—	20	—	20
30	Deep	—	5	—	22
31	Deep	—	21	—	15
32	Surface	0	5	1	22
33	Surface	1	5	0	24
34	Surface	1	3	1	34
35	Deep	0	3	0	20
36	Deep	0	—	6	—
37	Deep	0	2	7	13
38	Surface	—	39	—	—
39	Deep	—	31	—	—
40	Surface	—	45	—	—
41	Deep	—	23	—	—
42	Surface	—	46	—	—
43	Deep	—	20	—	—

TABLE III. *The formation of crystalline precipitates after four weeks on agar media during the development of organisms isolated from lake muds.*

Media	Number of cultures tested	Number showing precipitates	Percentage showing precipitates
Cultures from surface mud			
Lipman's	79	62	78
Ca sulphate	81	42	52
Mud extract	78	18	23
Molisch's	83	0	0
Cultures from deep muds			
Lipman's	86	64	74
Ca sulphate	88	37	42
Mud extract	84	1	1
Molisch's	92	1	1

taining calcium were not observed to precipitate calcium carbonate. Only a few such colonies were observed.

Selected aerobes from lake muds.— Total aerobic plate counts were made on sodium caseinate agar of the lake muds. From these plates about 180 colonies were picked and inoculated into plates of calcium sulphate, calcium lactate, mud extract, and Molisch's agar media. The number and percentage of colonies from the surface and deep muds respectively that formed crystals after four weeks at 28°C are shown in Table III. The crystals found in the mud extract and Molisch's media were so small and few in number that no effervescence was observed on addition of hydrochloric acid. The only source of calcium in these media is the lake water with which they were made.

Common aerobic organisms.–Streak cultures on calcium sulphate and calcium lactate agar were made of some of the common aerobic organisms in stock in the laboratory. The growth was examined under the microscope at intervals for a period of four weeks. Table IV indicates the time at which crystals were observed within the colonies or in the surrounding media.

Common anaerobic organisms.— Streak cultures of *Clostridium botulinum*, *Cl. multifermentans*, *Cl. butyricum*, *Bacillus macerans*, and several

TABLE IV. *Time required for calcium carbonate crystals to appear in colonies of various aerobic organisms on calcium sulphate and calcium lactate media.*

Organisms	$CaSO_4$ agar days					Ca lactate agar days				
	3	7	14	21	28	3	7	14	21	28
Azotobacter chroococcum	−	+	+	+	+	−	−	−	−	−
Rhizobium leguminosarum	−	−	−	−	−	−	−	−	−	+
Staphylococcus aureus	−	−	−	−	−	−	−	−	−	−
Sarcina lutea	+	+	+	+	+	−	−	−	−	−
Serratia marsescens	+	+	+	+	+	−	−	+	+	+
Serratia fuchsina	+	+	+	+	+	+	+	+	+	+
Pseudomonas fluorescens	+	+	+	+	+	−	−	−	−	−
Pseudomonas phosphorescens	−	+	+	+	+	−	−	−	−	−
Achromobacter hartlebii	−	+	+	+	+	−	−	+	+	+
Escherichia coli	+	+	+	+	+	−	−	−	+	+
Escherichia communior	+	+	+	+	+	−	−	−	−	−
Escherichia acidilactici	+	+	+	+	+	−	−	−	−	+
Aerobacter aerogenes	+	+	+	+	+	−	−	−	−	+
Proteus vulgaris	+	+	+	+	+	−	+	+	+	+
Proteus mirabilis	+	+	+	+	+	−	−	−	−	−
Proteus ichthyosmius	−	−	−	−	−	−	−	−	−	+
Alcaligenes viscosus	+	+	+	+	+	−	−	−	−	−
Bacillus subtilis	−	−	+	+	+	−	−	+	+	+
Bacillus graveolens	+	+	+	+	+	−	+	+	+	+
Bacillus megatherium	−	+	+	+	+	−	−	+	+	+
Bacillus mesentericus	−	−	−	+	+	+	+	+	+	+
Bacillus fusiformis	+	+	+	+	+	−	−	−	−	−
Oidium lactis	−	+	+	+	+	−	−	−	−	−
Mycoderma monosa	−	−	−	+	+	−	−	−	−	−
Saccharomyces cerevisciae	−	−	−	−	−	−	−	−	−	−
Saccharomyces ellipsoideus	−	−	−	−	−	−	−	−	−	−

anaerobes isolated from the deposits of Lake Mendota were not observed to precipitate calcium carbonate after seven days at 37°C. Each of the above cultures were incubated under anaerobic conditions using Bacto-beef agar, modified Speakman's agar medium and a one per cent tryptone agar containing one-tenth of one per cent glucose. Each medium contained 180 milligrams of soluble calcium per liter added as the chloride.

The above experiments indicate that many different classes of aerobic organisms will precipitate calcium carbonate. The possible role of micro-organisms in the precipitation of the crystals in nature should not therefore be viewed with an eye to one or two species of bacteria as has been done by several investigators. Throughout these experiments it was noticed that the ability of certain organisms to precipitate the carbonate is quite erratic. Thus parallel plates of mud dilutions in the same media showed wide differences in the percentage of colonies forming crystals. Also it was often three or four weeks before crystals appeared in certain colonies although good growth was secured after three to four days. The percentage of the aerobic carbonate-precipitating organisms from lake muds developing on agar plates varied from 0 to 70 per cent.

EFFECT OF THE CONCENTRATION OF CALCIUM ON THE PRECIPITATION OF THE CARBONATE

It appeared from the work of Lipman and Molisch that it would be desirable to determine the concentrations of calcium necessary in artificial media for precipitation before attempting to demonstrate it as occurring under natural conditions.

Molisch's agar medium was prepared and to different portions were added varying amounts of calcium chloride. The soluble calcium in the series ranged from 3 to 200 milligrams per 100 cubic centimeters. Plates were poured of these media and were streaked with organisms from lake muds and stock cultures which would precipitate calcium carbonate from a calcium sulphate medium. Table V indicates uniformly positive precipitation in the higher concentrations of calcium and that many of the organisms can cause precipitation in a medium containing only ten milligrams of calcium per 100 cubic centimeters. It was necessary as above to incubate for at least four weeks. *Proteus vulgaris* was found to precipitate crystals in a medium which contained only the amount of calcium found in lake waters. The crystals were, however, too small and few in number to detect effervescence with hydrochloric acid. Inasmuch as no precipitation was observed at the next higher concentration of calcium by this organism, it is doubtful whether these crystals were carbonate. Inspection of the table, however, shows that skips occurred in several other instances; *e.g.*, *E. communior*, 13c–a and 13x.

In order to demonstrate further the effect of the concentration of calcium on the ability of the organisms to precipitate the carbonate, the fol-

TABLE V. *Appearance of $CaCO_3$ crystals in Molisch's medium containing various amounts of soluble calcium.*

Organism	Time in Days								
	Milligrams of Ca per 100 cc. medium								
	200	150	100	75	50	35	20	10	3.3
4a	4	4	4	4	4	4	14	14	—
4c	4	4	4	4	4	7	7	21	—
6	14	14	14	14	14	14	21	28	—
10a–c	—	—	—	—	—	—	—	—	—
10b–b	4	4	4	4	4	4	7	21	—
10b–c	4	4	4	4	4	4	7	21	—
11a	14	14	14	14	14	14	21	—	—
12a	4	4	4	7	7	14	14	—	—
12c	4	4	4	7	14	14	—	—	—
13c–a	—	—	21	21	28	—	—	—	—
13x	—	—	—	—	21	—	—	28	—
Serratia fuchsina	4	7	7	7	7	7	7	—	—
Sarcina lutea	4	7	14	14	14	14	14	21	—
Pseudomonas fluorescens	7	14	14	14	28	14	21	—	—
Alcaligenes viscosus	14	14	14	14	14	14	28	—	—
Escherichia acidilactici	14	14	14	14	14	14	28	—	—
Escherichia communior	14	14	14	—	21	21	28	28	—
Aerobacter aerogenes	14	14	14	14	21	21	—	—	—
Proteus vulgaris	7	7	14	14	14	14	14	—	21
Bacillus graveolens	14	14	14	14	14	14	21	—	—

lowing experiment was carried out.

Eighteen bottles, each containing 1200 cubic centimeters of Molisch's medium with various concentrations of soluble calcium as the chloride, were set up in triplicate. Two of each concentration were inoculated with two actively precipitating organisms isolated from lake muds (4c and 10b–b), while the third was left as control. Analyses of the media for soluble calcium were made periodically on all bottles. The results are indicated in Figure I. The method used on all calcium determinations was precipitation as the oxalate and titration with permanganate. The apparent increase in soluble calcium in the control bottles was due to evaporation. Thus the true amount of precipitation in the inoculated bottles is somewhat greater than is indicated by the graph. In one of the control bottles contamination from the air occured during sampling. A subsequent decrease of the calcium in solution was observed due to carbonate precipitation by the contaminant.

These curves indicate that often precipitation did not occur until after the fourth day, or until growth was initiated, and in some instances not until after the thirty-fifth day. In the lower concentrations of calcium we find a marked precipitation in some of the bottles but not in others. The lowest curve on each graph represents those bottles to which no additional calcium was made. In one instance the calcium concentration fell below that initially lower curve. Each of the points on these graphs however are determined only by a single analysis.

The peptone used in all of the media was tested qualitatively for calcium both before and after ashing. None could be detected.

From the above we can safely conclude that certain organisms are capable of precipitating calcium from solutions that contain ten milligrams of calcium per 100 cubic centimeters. This concentration is less than one quarter of that found in open sea water. We also have some evidence that microorganisms are capable of forming carbonate crystals in liquid and agar media containing only that amount of calcium found in the waters of Lake Mendota.

ANALYSES OF THE CRYSTALS

Crystals that had been precipitated by pure cultures of microorganisms in liquid calcium sulphate and calcium lactate media and Molisch's medium plus calcium chloride were separated and freed from organic matter as much as possible by treatment with dilute alkali and thirty per cent hydrogen peroxide. In order to determine whether the crystals were actually calcium carbonate, the ratio of calcium present to carbon dioxide liberated on treatment with hydrochloric acid was determined. The results of these analyses of the crystals from the different media are given in

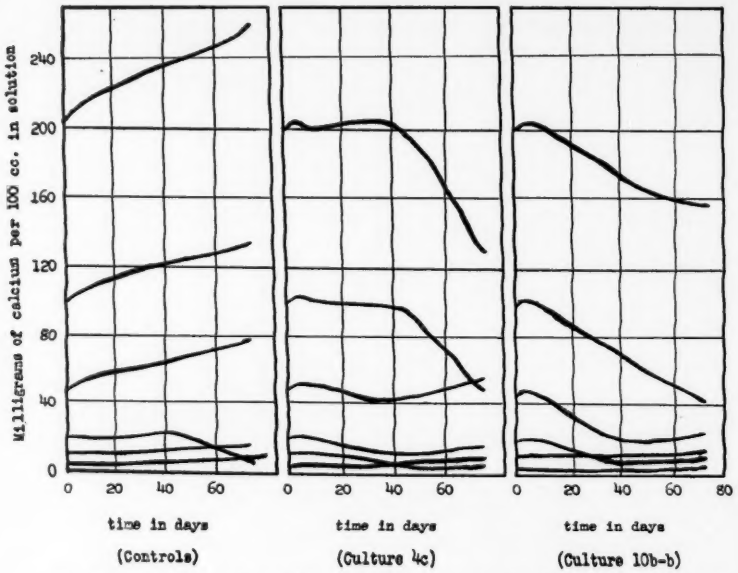

FIG. 1. Rate of Decrease in Soluble Calcium in Molisch's Medium when Inoculated with Pure Cultures of Calcium Carbonate Precipitating Organisms

Table VI. From these it may be assumed that the crystals are nearly pure calcium carbonate. None of the crystals gave a positive qualitative test for phosphates.

X-ray patterns of the crystals show that the carbonate is in the form of calcite. The deep mud from the bottom of Lake Mendota also consists chiefly of calcite as determined by X-ray pattern.

ARE MICROORGANISMS RESPONSIBLE FOR THE PRECIPITATION OF CALCIUM CARBONATE IN NATURE?

It has been shown that the precipitation of calcium carbonate by certain microorganisms may be demonstrated very easily under artificial conditions. Two attempts have been made by the authors to demonstrate precipitation under natural conditions such as found in the deposits of Lake Mendota.

Several "glasine" (similar to viscose) sacks were prepared and fitted tightly by means of rubber bands and autoclaving over the ends of one inch test tubes from which the bottoms had been removed. The other ends of the tubes were closed by means of rubber stoppers. This membrane would allow the passage of soluble substances such as organic salts, sugars and other nutrients but was impervious to bacteria.

After autoclaving under water, four of the containers were inoculated with dilutions of mud and were sealed while three others were sealed sterile. Each was placed in an individual casing which would allow diffusion from the outside and lowered separately to the bottom of Lake Mendota in about 80 feet of water. They were allowed to incubate in the mud for six weeks. On removal to the laboratory it was noted that some growth had occurred within the inoculated containers while the controls remained sterile. The material in each was examined microscopically but no crystals of any kind were observed either in the liquid or adhering to the "glasine" membrane. This experiment was thus negative as indicating precipitation within the lake muds by microorganisms.

The second experiment consisted of placing two liters of fresh lake mud and six liters of lake water into each of nine ten-liter Pyrex bottles. Six of these were autoclaved for eight hours and three left unsterilized. Three of the first six were left uninoculated, while the other three were inoculated with 100 to 200 grams of fresh raw

TABLE VI. *Analyses of crystals precipitated by microorganisms from calcium-bearing media.*

Media	Weight of sample	Calculated calcium carbonate in crystals from		Ca/CO_2 ratio
		(a) CO_2 liberation	(b) Ca determination	
	gms.	gms.	gms.	
Calcium sulphate	0.315	0.301	0.283	0.94
Calcium lactate	0.315	0.309	0.299	0.97
Molisch's+$CaCl_2$	0.315	0.298	0.284	0.95

lake mud. One of each group of three was incubated without additional calcium; in the second and third bottle of each group the amount of soluble calcium was doubled and tripled respectively by the addition of calcium chloride. This series was incubated at room temperature for a period of 358 days. At various intervals samples were removed and plate counts on sodium caseinate agar and pH readings were made on each bottle. The samples were then passed through a Sharples super-centrifuge and the amount of calcium determined for each sample in duplicate. The bottles were stoppered with cotton and covered with lead foil so that evaporation would not occur to any appreciable extent. The results are given in Table VII and Figure 2.

A duplicate series was prepared in which the bottles were tightly stoppered and allowed to incubate at the bottom of Lake Mendota for 166 days. After this period they were incubated an additional 163 days at room temperature. These results are presented in Table VIII and Figure 3.

It will be noted that in both series the soluble calcium increased markedly at the beginning of the incubation period. This initial rise may be attributed in part to the production of carbon dioxide within the bottles causing the formation of the soluble bicarbonate of calcium from the carbonate present in the mud. In several instances this increase occurred in the controls before contamination was evident. It was very difficult to keep the controls sterile when sampling due to their size. Thymol was added eventually to each control bottle in an attempt to hold down growth.

From the tables and graphs there does not appear to be any correlation between the rate of growth of microorganisms, reaction, or the soluble calcium content of the mud. The rise in calcium was followed by a decrease in most instances. After nearly a year the amount of calcium had not decreased to its original value. Even those bottles that carried three times as much calcium in solution as found in lake waters do not approach the lower value at all. In the case of the unsterilized mud it will be noted that more calcium is apparently coming into solution even after a year's time. In these instances the initial rise was not nearly so marked, however, as in those bottles that were autoclaved.

When the samples of the room temperature bottles were taken after 188 days and the lake temperature bottles after 166 days, dilution plates were made on Molisch's medium plus one half of one per cent calcium chloride. After four weeks of incubation the plates were examined and from one to ten per cent of the colonies thereon were observed to precipitate calcium carbonate crystals. Qualitative tests for the presence of ammonia and nitrites in the mud solutions were also made at the above time. Neither could be detected in any of the bottles.

It would be expected, if microorganisms are capable of precipitating calcium carbonate under lake conditions, that we should have an indication of this precipitation in the fore-

TABLE VII. *Number of aerobic organisms in mud bottles and reaction at various intervals—temperature—28° C.*

Bottles	Days	1-x Calcium in lake water		2-x Calcium in lake water		3-x Calcium in lake water	
		Number of bacteria per cc.	pH	Number of bacteria per cc.	pH	Number of bacteria per cc.	pH
Controls	0	Sterile	7.8	Sterile	7.7	Sterile	7.8
	14	Sterile	7.8	Actino.	7.8	Actino.	7.8
	42	Molds	8.2	Actino.	8.2	Actino.	8.2
	105	Actino.+ molds	8.4	Actino.	8.3	Actino.	8.3
	188	Actino.+ molds	7.7	Actino.+ molds	7.6	Actino.+ molds	8.2
	358	Actino.+ molds	7.5	Actino.+ molds	7.3	Actino.+ molds	6.9
Sterilized and raw mud	0	18,600	7.8	21,500	7.7	26,000	7.7
	14	30,000,000	8.3	10,900,000	8.0	27,200,000	8.2
	42	4,800,000	8.3	4,600,000	8.4	3,900,000	8.4
	105	5,000,000	8.4	5,000,000	8.2	4,000,000	8.3
	188	5,200,000	7.8	7,700,000	7.6	4,500,000	7.6
	358	1,630,000	7.5	1,460,000	7.6	1,300,000	7.2
Unsterilized raw mud	0	590,000	7.8	700,000	7.4	1,230,000	8.2
	14	780,000	8.4	1,000,000	8.2	650,000	8.2
	42	670,000	8.4	610,000	8.4	680,000	8.3
	105	179,000	8.2	1,430,000	8.3	240,000	8.2
	188	820,000	7.8	600,000	7.7	590,000	7.8
	358	390,000	7.3	410,000	7.2	310,000	7.3

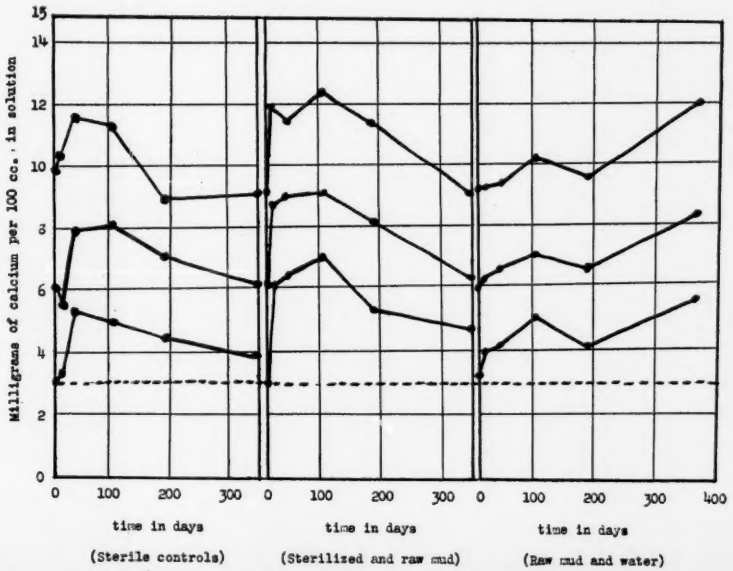

FIG. 2. Soluble Calcium in Lake Mud and Water at Various Times. Room Temperature.

TABLE VIII. *Number of aerobic organisms in mud bottles and reaction at various intervals—lake temperature.*

Bottles	Days	1-x Calcium in lake water		2-x Calcium in lake water		3-x Calcium in lake water	
		Number of bacteria per cc.	pH	Number of bacteria per cc.	pH	Number of bacteria per cc.	pH
Controls	0	Sterile	7.8	Sterile	7.7	Sterile	7.9
	84	Sterile	8.2	Slightly contaminated	7.4	Slightly contaminated	7.8
	166	Approx. 100 (molds, bact.)	6.8	Approx. 500 (bacteria)	6.7	Approx. 20 (bacteria)	6.6
	328	Approx. 15,000 (molds, bact.)	6.4	Less than 1,000	6.4	Approx. 15,000 (molds, bact.)	6.4
Sterilized and raw mud	0	89,000	7.8	106,000	7.6	62,000	7.5
	84	1,340,000	7.6	1,330,000	7.3	Broken	—
	166	2,100,000	6.8	2,000,000	6.6	Broken	—
	328	3,700,000	6.4	3,500,000	6.4	Broken	—
Unsterilized raw mud	0	710,000	8.2	860,000	8.0	490,000	8.2
	84	Broken	—	347,000	8.2	2,350,000	8.0
	166	Broken	—	960,000	6.8	1,900,000	6.7
	328	Broken	—	600,000	6.4	800,000	6.5

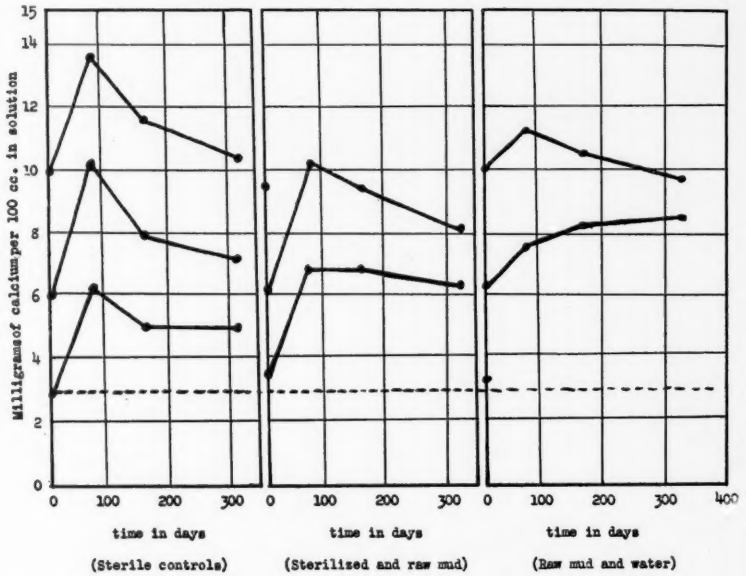

FIG. 3. Soluble Calcium in Lake Mud and Water at Various Times. Lake Bottom Temperature.

going experiment. This precipitation should have been apparent at least in the bottles to which soluble calcium was added. As was explained before the formation of carbon dioxide within the bottles leads to an increase in soluble calcium. Thus the formation of carbon dioxide might tend to mask any carbonate precipitation within the bottles. If precipitation had occurred, however, it would be expected that the final concentration of calcium within each series would be very nearly identical, even though not at that level found in natural lake waters.

SUMMARY

It has been shown that many common aerobic organisms will precipitate calcium carbonate in pure culture under artificial conditions. Many such organisms may be isolated from the deposits of Lake Mendota, a fresh water lake in Wisconsin. Several cultures of aerobic organisms were observed that would precipitate calcium carbonate crystals from a suitable medium containing as little as ten milligrams of soluble calcium per 100 cubic centimeters. Some evidence was obtained that crystals could be produced from media containing only the amount of calcium in lake water or approximately three milligrams per 100 cubic centimeters. Anaerobic organisms or colonies of organisms developing on plates of calcium-bearing media incubated under anaerobic conditions were not observed to precipitate the carbonate in any instance. Colonies of algae did not form crystals in the few instances observed.

The crystals produced by pure cultures from various media were analyzed and found to be nearly pure calcite, the form of calcium carbonate present in the lake mud.

Two experiments were carried out, the results of which have failed to prove connection of the microorganisms and calcium carbonate precipitation under natural conditions such as found in the deposits of Lake Mendota.

The authors are indebted to E. B. Fred for his helpful criticism and advice and to W. H. Twenhofel for his constant interest and help throughout this work.

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A ROTARY TYPE OF SAMPLE SPLITTER

C. K. WENTWORTH, W. L. WILGUS AND H. L. KOCH

ABSTRACT

The writers discuss certain problems of sampling and describe tests of a rotary splitting machine in a large and a small form. Used on sandy gravel for mechanical composition samples the rotary device shows marked superiority over the Jones type. Used on mineral grain mixtures, where the size of final sample is purposely restricted the rotary machine gives results perhaps slightly better than those from non-rotary devices but it is doubted if much more valid splitting can be expected from any mechanical device because of the limit inherent in statistical theory as applied to small samples.

The problem of collecting samples, or parts of a whole, the properties of which are studied as representing the whole, is an ancient one, troublesome alike to students of natural and physical sciences as well as of the social sciences. And because of the need, in many instances, for economy of time and for using the smallest sample which will be sufficiently representative, the problem of "the small sample" is a classic one among students of statistical methods.

Both in the social and the physical sciences, we encounter the phenomenon of linked properties or characteristics, whereby it becomes nearly impossible to select a normal sample. In the social field this is one of the evils of "questionairing" and in the physical field a host of behavior-controlling characteristics, such as density, electrical properties, shape, sizes of units and the like, spring up to defeat the segregation of normal samples by mechanical methods.

In the field of geology, because of the magnitude and inaccessibility of large parts of the rock units dealt with, the necessity for study by sampling is evident. Much geologic field work is merely formation sampling, using outcrops as units of selection. Necessarily such sampling is very crude, viewed statistically, since the selection is done commonly on an opportunist rather than a random or equispaced basis. The latter method has, however, been used for certain types of areal geologic work. Among economic geologists and mining engineers, mine and mineral property sampling are common practices and most analyses of rock materials are based on some sort of sampling procedure, both in the field and also in the use of finely powdered material for the components of the analysis. Sedimentationists encounter the sampling problem especially in dealing with detrital sediments which are subjected to mechanical, shape,

lithologic, or density analysis. Because sediments are studied for their inherent size and shape distributions, it is not possible to split the sample by crushing to standard size, as in the case of coal samples or chemical analysis samples. The latter, being essentially homogeneous physically, can be split by simple quartering into fairly valid fractions, but it is well known that in sand or gravel samples, where the particles vary in size, shape and density, almost every pouring, shaking, or stirring treatment to which they may be subjected tends to develop inhomogeneities related to top, bottom or sides of containers. These tendencies have to be very adroitly combatted in order to maintain and split off normal samples.

The best known device is the Jones sample splitter, consisting of a system of chutes leading alternately to the two pans which are placed beneath. In principle this apparatus is fairly effective if the number of chutes is large, even, and if the pouring is done with care from a scoop having exactly the width of the chute system. But that in practice the machine may be badly operated is suggested by the fact that the writer once purchased from an instrument company a Jones splitter accompanied by a scoop at least one chute width wider than the chute system. Pouring sediment into the funnel neck above the chutes, such a scoop has possibilities for the most heinous vitiation of any splitting process, and if makers misunderstand underlying principles, so are users likely to do so.

The possible right- or left-handed vitiation of splitting in a bilateral splitter may be progressively reduced by increasing the number of alternate chutes constituting the system so long as these remain relatively large in comparison to the size of the particles to be passed. When the sizes of particles approach the chute width, so that bouncing of particles across one or more chutes becomes common, further suspicions are raised as to the strict normality of the scoop stream and the symmetry and homogeneity of the end zones. Thus any great increase in the number of chutes in order to combat possible right- or left-handed asymmetry in the scoop stream is likely to introduce other, and still less known complications, and hence appears impracticable.

Recently several students have approached the problem of splitting heavy mineral concentrates so as to obtain normal fractions for mounting on a microscope slide. Krumbein used a funnel so set over a container divided into sectors by knife-edges that the funnel stream was split into nearly equal, and presumably normal fractions.[1] Otto,[2] following Krumbein, devised the "Microsplit" which is essentially a well made, precise Jones splitter. Pettijohn[3] has also devised a method of splitting.

[1] The instrument and method are described by G. H. Otto on pages 31 and 32 of the paper cited below.

[2] Otto, G. H., Comparative tests of several methods of sampling heavy mineral concentrates: *Jour. Sed. Petrology*, vol. 3, no. 1, pp. 30–39, 1933.

[3] Pettijohn, F. J., The petrography of the beach sands of Southern Lake Michigan: *Jour. Geology*, vol. 39, footnote, p. 436, 1931.

Each of these methods involves closer attention to mechanical details of methods already in use, and each appears to improve the validity of the small samples split off. However, in none of these methods has a fundamental improvement of the underlying theory been made, nor does any involve an expansion of the accuracy possible under well-known statistical principles. It has long been the view of the senior author that modification and expansion of the statistical conditions would be more fruitful in accuracy and freedom from instrumental imperfections and personal equations. The elimination of possible right- and left-handed asymetries of the Jones splitter seemed to require a rotary type of splitter in which the time irregularities of the whole stream are substituted for the geometrical non-homogeneity of the usual stream, and in which the latter can be split into numerous fractions and redistributed into alternate portions by using a rapid rotation and a slow rate of flow. This is essentially the principle of many devices used in ore-treatment mills for continuous collection of small samples of run-of-mine or run-of-mill products.

Recent occasion for splitting both

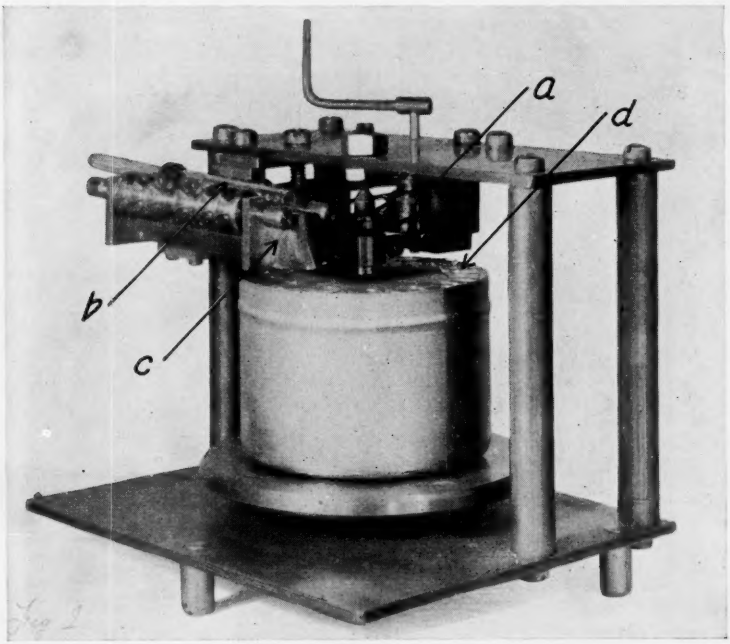

FIG. 1.

mechanical analysis samples and mineral composition samples encouraged the construction of two rotary splitting machines and with the assistance of the junior authors each in connection with work on a previously chosen analytical problem, the following tests and comparisons were made.

DESCRIPTION OF THE ROTARY SPLITTERS

The two rotary splitters are of similar design, one for large samples of coarser material and the other for small samples of heavy minerals and the like. The mega-splitter consists of a ply-board turntable mounted on a vertical steel axle whose hardened points pivot in adjustable center screws. On the turntable are mounted sixteen, four-ounce metal pill boxes in a circle concentric with the axle. A funnel is arranged directly above this circle so that as the turntable spins the boxes pass under the funnel in turn (see Figure 1). By adjusting the rate of turning, by twirling the axle with the fingers, to the rate of flow through the funnel each box may be made to pass as many times as desired under the funnel stream.

With the rotary machine two methods are available in splitting fractions from a sample. One may either directly accept the sixteenths established at the first split, or eighths formed by combining opposite sixteenths, or quarters by each fourth sixteenth or he may halve the sample by taking alternate sixteenths and by repeating this operation, halve the sample successively down to quarters, eighths or smaller fractions as desired. The former method is faster; the latter has a larger guarantee of accuracy against irregularities of operation.

In the micro-splitter, the arrangement of parts is similar. A circle of sixteen small glass vials was used instead of the pill boxes. The chief difficulty in the microsplitter was in producing a slow, fairly steady and nearly random stream of sand grains so as to prolong the flow of a very small sample. This was accomplished by mounting a small section of glass tube on a pair of trundle rollers which were driven by a rubber band belt from a small hand crank. The turntable was driven by another belt from the same crank. The angle of tilt of the rollers and the glass tube is adjustable so as to regulate the rate of discharge of sand grains from the lower, open end of the tube. (See Figure 1) It was found that a small funnel with a tangentially elongated outlet reduced the loss of grains by bouncing from the edges of the moving vials. In both the mega and microsplitters, improvements can easily be made in certain parts of the design, especially in using containers which are sector-shaped rather than round so as to reduce the loss by bouncing and imperfect juxtaposition of the containers.

TESTS OF THE ROTARY MEGA-SPLITTER

Four samples of essentially similar material were split, two in a Jones splitter to quarters, one in the rotary splitter to quarters by successive

halving and one in the rotary splitter to eighths. All these fractions were mechanically analysed in a Rotap shaker, using a ten-minute shaking period. The results of these tests are shown in the following tables.

The data carried in Tables I, II and III are also shown in the graph of Figure 2. It is evident from the average deviations shown in the several tables and the sub-joined general averages that the fractions split off by the rotary method show much less variation among themselves and a

TABLE I. *Sample No. 1, quartered by Jones splitter.*

Approximate grade size (millimeters)	Grade no.	Average cumulative per cent[a]	Deviations Units=0.0001 of whole				Arithmetic mean deviation	Mean deviation, relative (unit =0.001 of next superior grade)
+2	1	2.35	−34	−52	+74	+12	43	183
+1.4	2	6.26	−69	−91	+122	+38	80	200
+1	3	12.71	−100	−116	+87	+129	108	167
+ .7	4	29.15	−215	−155	+145	+225	185	112
+ .5	5	46.84	−248	−134	+186	+196	191	108
+ .35	6	59.53	−183	−135	+146	+172	159	125
+ .25	7	74.95	−155	−95	+105	+145	125	81
+ .175	8	88.27	−87	−77	+72	+92	82	62
+ .125	9	97.68	−18	−18	+12	+24	18	19
+ .88	10	99.70	−7	−3	+4	+6	5	25
+ .62	11	99.985	−.5	−.5	+.5	+.5	.5	18
							Average 91	Average 100

[a] In various tables and text certain averaged and other values are given to the nearest unit; decimal fractions resulting from computing process are omitted.

TABLE II. *Sample No. 2, quartered by Jones splitter.*

Grade no.[a]	Average cumulative per cent	Deviations Units=0.0001 of whole				Arithmetic mean deviation	Mean deviation, relative (unit =0.001 of next superior grade)
1	2.06	+15	−10	+1	−6	8	39
2	6.32	+57	−25	−44	+12	35	82
3	13.98	+99	−59	−75	+35	67	87
4	32.00	+140	−35	−135	+30	85	47
5	50.03	+147	−1	−147	+1	74	41
6	62.52	+146	−2	−152	+8	77	62
7	77.25	+105	+5	−125	+15	63	43
8	89.53	+47	−3	−51	+7	27	22
9	97.76	+19	−66	+33	+14	33	40
10	99.70	−4	−3	+8	−1	4	20
11	99.96	−1	−1	−1	+3	1.5	60
						Average 43	Average 49

[a] In this and succeeding tables grade sizes are the same as in Table I.

TABLE III. *Sample No. 3, quartered by rotary splitter.*[a]

Grade no.	Average cumulative per cent	Deviations Units=0.0001 of whole				Arithmetic mean deviation	Mean deviation, relative (*unit=0.001* of next superior grade)
1	2.56	+19	−12	−8	+1	10	40
2	6.28	−4	+31	−17	−10	15	40
3	12.90	−44	+47	+1	−4	24	36
4	29.02	+17	+19	−24	−12	18	11
5	46.84	+6	+22	−24	−4	14	8
6	59.26	+14	+18	−26	−6	16	13
7	74.55	−5	+7	−5	+3	5	3
8	87.95	−5	−5	+5	+5	5	4
9	97.46	−6	−6	+4	+8	6	6
10	99.64	−4	−2	+2	+4	3	14
11	99.97	−2	+1	−1	0	1	30
						Average 11	Average 19

[a] Sample first split to half and each half then resplit. Split to halves in nine revolutions. Each half = 72 of 144 samples taken alternately in time. Quarters established in five revolutions. Each quarter = 40 of 80 samples taken alternately in time.

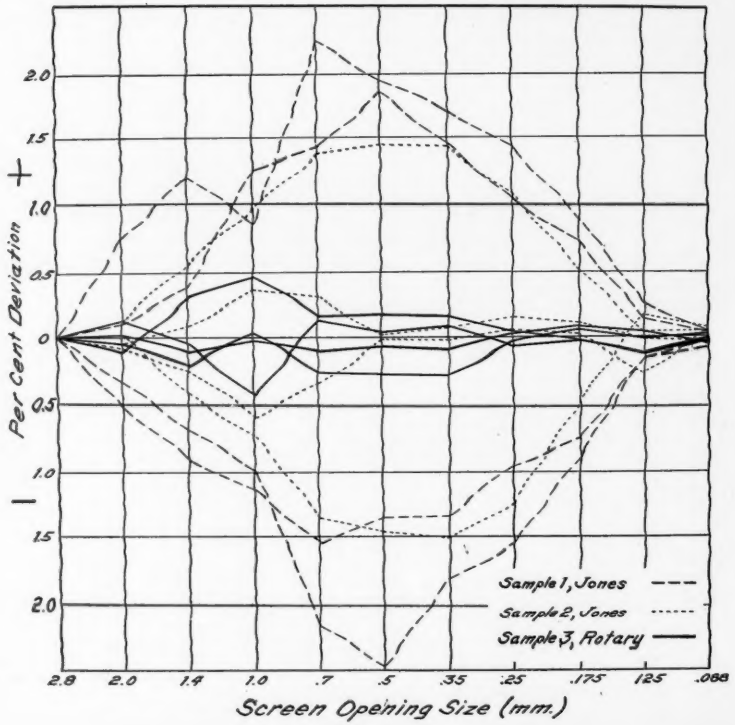

FIG. 2.

much closer approach to the normal or average composition than do those split in the Jones splitter.

The absolute average deviations of the two Jones tests are 0.91 per cent and 0.43 per cent, averaging 0.67 per cent. Those of the two Rotary quarterings, by two different methods are 0.11 per cent and 0.10 per cent respectively, less than one-sixth of the average error in the Jones operation. Using the deviations relative to next superior grades the ratio is only slightly less favorable to the Rotary splitter. Comparison of the data of Tables IV and V shows that the average error in the several grades of the various eighths is about $2\frac{1}{2}$ times as great as the error of the grades in the quarters, as would be expected. But errors, even in the eighths established in one operation by the Rotary method are less than the average or even the most accurate of the quarterings accomplished in the Jones splitter.

TESTS OF THE ROTARY MICRO-SPLITTER

Chief need of micro-splitting appears in taking valid small samples of heavy mineral fractions; hence this device was tested on a prepared mixture of readily identifiable mineral grains. All grains were between $\frac{1}{4}$ and $\frac{1}{2}$ mm. in diameter, except a few of the corundum (emery) grains, which were smaller. The entire mixture was split into working fractions in the Rotary splitter; it is not, however, assumed that the material used in the two tests is identical. Rather, results on the two machines are compared with their own averages, since all fractions were counted.

Table VI shows the count on four quarters established in the Jones splitter and Table VII similar data

TABLE IV. *Sample No. 4, quartered in rotary splitter.*[a]

Grade no.	Average cumulative per cent	Deviations Units = 0.0001 of whole				Arithmetic mean deviation	Mean deviation, relative (unit = 0.001 of next superior grade)
1	2.19	+10	−5	+18	−23	14	64
2	5.47	+3	+2	+11	−16	8	24
3	11.60	−11	−1	+11	+1	6	10
4	27.06	−16	−13	+40	−11	20	13
5	44.63	−30	+7	+37	−14	22	13
6	57.25	−27	+7	+25	−5	16	13
7	73.40	−10	+9	+11	−1	10	6
8	87.30	−5	+10	0	−5	5	4
9	97.21	−1	+4	−2	−1	2	2
10	99.50	0	−1	−1	+2	1	4
11	99.96	−1	−2	+2	+1	1.5	20
						Average 10	Average 16

[a] Directly by combining every fourth 16th.

TABLE V. *Sample No. 4, divided into eighths in rotary splitter.*[a]

Grade no.	Average cumulative per cent	Deviations Units=0.0001 of whole								Arithmetic mean deviation	Mean deviation, relative (unit =0.001 of next superior grade)
1	2.23	−35	−4	−19	−14	+46	+17	+48	−39	28	125
2	5.47	−49	−20	−2	+39	+57	+25	+23	−73	36	111
3	11.60	−57	+5	−4	+45	+34	−5	+24	−42	27	44
4	27.16	−37	+39	+64	+25	−16	−78	+4	−1	33	21
5	44.70	−39	+55	+80	+69	−10	−50	−5	−100	51	30
6	57.24	−34	+46	+63	+51	−14	−24	−24	−64	40	32
7	73.39	−9	+41	+48	+31	−14	−9	−19	−69	30	19
8	87.36	−16	+14	+4	+14	+14	+14	−6	−38	15	11
9	97.20	0	+30	0	+10	0	+10	−20	−20	10	10
10	99.49	+11	−4	−19	+1	+8	+11	−9	+1	8	35
11	99.97	−2	−1	−1	−1	+2	+1	+1	+1	1.25	26
										Average 26	Average 42

[a] Directly by combining opposite 16ths, same operation as table IV. Split in 15 revolutions. Each eighth = 30 of 240 samples taken at regular intervals.

for quarters established directly on the Rotary splitter by combining 16ths in proper rotation.

Since the rotary separation shown as quarters in Table VII was made in sixteenths and counted in this form it was possible to compute it as sixteenths, eights and halves, as well as the quarters shown above. The detailed tabulation is omitted; the computed and experimental probable errors and the ratios between them are shown in Table VIII. The computed probable error is computed by means of the usual formula, as given by Dryden.[4] The experimental probable error is computed from the standard deviation (square root of mean of squared deviations) by multiplying by the constant 0.6745. According to Table VIII the experimental results are slightly more accurate than statistical theory would assure. This is of course accidental and it is evident that the average ratio derived is a very crude result of combining in equal weight a small number of determinations of unequal validity. To derive a reliable measure of approach of the rotary machine to the accuracy justified by theory would be a very tedious process but the fairly close approach indicated by the several results suggests that not only the rotary machine but the Jones splitter is here also approaching somewhat closely to the theoretically indicated value.

Tables IX shows the results of an additional comparison between the two splitters. In the rotary splitter the fractions were established by successive splitting to halves. An effort was made to adjust the two samples to yield the same totals and a part of the sample split in the rotary machine was poured back from a tube. This resulted in the abnormal proportions of olivine and corundum

[4] Dryden, A. L., *Op. cit.*, p. 235.

TABLE VI. *Mineral mixture quartered by Jones splitter.*

Mineral	Total no. of grains (4 quarters)	Average per cent	Deviation (units=0.0001 of whole of each quarter=approximately 1/5 grain)	Mean deviation	Mean deviation, relative (unit=0.001 of fraction)	Computed P.E. in per cent of each quarter	Experimental P.E. in per cent of each quarter	Ratio exper. to computed P.E.
Quartz	4672	57.57	+ 86 +88 +82 −256	128	22	.74	1.00	1.35
Black Glass	2620	32.26	+ 40 +52 − 2 − 90	46	14	.69	.37	.54
Olivine	457	5.65	−168 −68 −56 +292	146	258	.35	1.17	3.35
Corundum	355	4.38	+ 35 −66 −20 + 51	43	98	.31	.31	1.00
Shell Debris	11	0.14	+ 6 − 4 − 4 + 2	4	286	.06	.027	.45
Total	8115		Averages	73	136	.43	.57	1.34

TABLE VII. *Mineral mixture quartered by rotary splitter.*

Mineral	Total no. of grains (4 quarters)	Average per cent	Deviation (units=0.0001 of whole of each quarter =0.15 grain—)	Mean deviation[a]	Mean deviation, relative (unit=.0001 of fraction)	Computed P.E. in per cent of each quarter	Experimental P.E. in per cent of each quarter	Ratio exper. to computed P.E.
Quartz	3377	56.76	−82 −108 +316 −126	158	28	.87	1.23	1.41
Black Glass	1941	32.65	+16 +204 −296 + 76	148	45	.81	1.23	1.52
Olivine	344	5.79	+43 − 83 − 33 + 73	58	103	.41	.41	1.00
Corundum	275	4.63	+18 − 7 + 4 − 15	11	24	.37	.08	.22
Shell Debris	10	0.17	+17 − 10 + 3 − 10	10	590	.07	.07	1.00
Total	5947		Averages	77	158	.51	.60	1.03

[a] In comparing these mean deviations with those of Table VI it should be noted that here the total number is only about three-fourths that of Table VI, hence the accuracy expected is considerably less, and that attained shows superior work of the rotary splitter.

TABLE VIII. *Comparison of computed and experimental errors.*

	Probable error computed	Probable error experimental	Ratio experimental to computed probable error
Sixteenths—Rotary			
Quartz	1.74	1.77	1.02
Black Glass	1.62	1.86	1.15
Olivine	0.82	0.85	1.04
Corundum	0.74	0.47	0.64
Shells	0.14	0.11	0.79
Eighths—Rotary			
Quartz	1.23	1.51	1.23
Black Glass	1.15	1.34	1.16
Olivine	0.58	0.51	0.88
Corundum	0.52	0.38	0.73
Shells	0.10	0.08	0.80
Quarters—Rotary			
Quartz	0.87	1.23	1.41
Black Glass	0.81	1.23	1.52
Olivine	0.41	0.41	1.00
Corundum	0.37	0.08	0.22
Shells	0.07	0.07	1.00
Halves—Rotary			
Quartz	0.61	0.81	1.33
Black Glass	0.57	0.91	1.60
Olivine	0.29	0.034	0.12
Corundum	0.26	0.067	0.26
Shells	0.05	0.051	1.02
			0.946

shown in this series as a whole. Here also the experimental P. E. is slightly less, on the average than the computed, though the exact figure is probably not significant.

DISCUSSION AND CONCLUSIONS

In the case of mechanical composition samples subjected to splitting by the Jones apparatus and the rotary splitter it seems quite clear from the tests that the rotary splitter achieves a large improvement in accuracy over the Jones splitter. It is plausible to suppose that the irregularity of composition of different parts of the stream fed to the small number of chutes of the Jones splitter leads to the variation shown in the corresponding fractions. On the other hand it is believed that in the use of the rotary splitter the time variations of the stream are less than those in the parts of the Jones stream and that such variations as remain are largely redistributed at random as the cups pass rapidly under the stream. Mathematical treatment of the factors involved in splitting off a normal mechanical composition fraction are vastly more complicated than in the case of mineral composition samples and has not been attempted. However, the number of

TABLE IX. *Comparison of the Jones and rotary splitters.*

	Mineral	Total no. of grains (8 eighths)	Average per cent	Deviations (units=0.001 of each eighth =approximately 2/3 grain)	Mean deviation	Mean deviation, (unit=0.001 of fraction	Computed P.E. in per cent of each eighth	Experimental P.E. in per cent of each eighth	Ratio exper. to computed P.E.
Jones Splitter	Quartz	2881	57.47	+ 9 −11 +11 +33 +26 −64 +17 −21	24	42	1.34	1.96	1.46
	Black Glass	1593	31.68	− 7 + 9 − 7 −29 +20 +66 −27 +15	23	73	1.27	1.96	1.54
	Olivine	357	7.10	− 5 + 8 + 1 + 9 − 6 − 3 − 6 + 2	5	70	0.67	0.40	0.60
	Corundum	142	2.80	+ 3 − 5 − 4 −13 − 2 + 3 +13 + 5	6	214	0.52	0.47	0.90
	Shell Debris	5	0.95	+ 1 + 1 − 1 − 1 0 − 1 + 2 − 1	1	105	0.25	0.07	0.28
	Total	4979			12	101			0.95
Rotary Splitter	Quartz	2795	56.30	+17 − 4 −35 − 9 +22 + 8 −30 +31	19	34	1.34	1.89	1.41
	Black Glass	1593	32.10	−19 − 4 +34 + 2 −12 − 6 +32 −27	17	53	1.27	1.42	1.12
	Olivine	283	5.84	− 3 +10 − 7 − 1 − 7 − 1 + 5 + 4	5	86	0.67	0.40	0.60
	Corundum	248	4.93	+ 5 − 3 + 9 + 8 − 4 − 2 − 6 − 7	6	122	0.52	0.40	0.77
	Shell Debris	4	0.83	− 1 + 1 + 1 − 1 + 1 + 1 − 1 − 1	1	120	0.25	0.07	0.28
	Total	4923			10	83			0.82

particles, at least in fine grades, is such as to impose no immediate statistical limit and to give theoretical ground for the large improvement effected by the rotary splitter. It is believed that still greater improvement could be achieved by extending the period of flow and increasing the number of turns of the machine, thus further utilizing the principle of repetition commonly applied in other kinds of measurements.

Splitting of sediment samples for mineral count, especially when the avowed purpose is to secure a valid sample consisting of a *small* number of grains offers greater difficulties. It should be recognized that here the probable error attainable can be readily computed from the number of grains and that even with complete freedom from instrumental irregularities this probable error cannot be reduced. Inspection of the deviations tabulated by Otto[5] and checking of the deviations shown in the tables above against computed values lead to the conclusion that all the various devices approach somewhat closely to the theoretical limit. Improvements of the better devices over the poorer must lie in the fairly narrow zone between the performance of the poorer machine and this limit and can hardly be very striking. With this slight range of possible improvements a very much longer series of tests than any yet carried out will be required to establish the superiority of a given machine. Theoretically the rotary machine should give better results; the present tests give some slight encouragement in this direction but do not amount to a demonstration.

Moreover, it is thought that theoretical limits to any substantial improvement of machine so long as numbers of grains remain small sets a point of diminishing returns for effort in this direction. The writer can only join Dryden in his plea that mineral grain counters state their results in such a form and with such data on total numbers as to indicate their statistical validity and that they count a sufficient number of grains as are necessary to reach the desired accuracy. The latter is, of course, entirely relative to the purpose of the work in hand and to the number and conformity of the samples dealt with. If time and materials permit, the counting of more basic samples is, after a given point, the road to more significant information, as opposed to mere accuracy.

[5] Otto, George H., *Op. cit.*, p. 37.

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DECEMBER, 1934

ARTIFICIAL OOLITES FROM SPRAY FROM AMMONIA-COOLING TOWER OF ICE PLANT, SEYMOUR, TEXAS

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ABSTRACT

The oolites form on the ground around sand grains by precipitation from the water sprayed over the ammonia-cooling tower at the ice plant in Seymour, Texas. This untreated water is hard, chiefly from calcium and magnesium bicarbonates, and precipitation is largely due to loss of carbon dioxide.

The oolites show two distinct concentric zones, in some of which four to nine laminae can be distinguished. No trace of radial structure was noted.

OCCURRENCE

The oolites described in this paper occur on the ground near the base of the cooling tower of the ice plant in Seymour, Texas. The pipes, containing hot compressed ammonia gas, are cooled by the water falling over them from the top of the tower. The untreated water used is city water obtained from shallow wells in the Seymour formation. The ice plant is in fairly constant operation in summer, and most of the oolites are found on the northwest side of the tower, due to the prevailing southerly winds.

DESCRIPTION

The oolites are only approximately spherical. Associated with them are small, irregular masses of sand and clay cemented by chemical precipitates. A few specimens show two to four oolites cemented together; another, a stalactite on a blade of grass.

The oolites are mostly less than 2 mm. in diameter. Of the one hundred and sixty measured with a vernier caliper, twenty-three exceeded 2 mm., and only five were less than 1 mm. in diameter. The table that follows gives the dimensions of the oolites that were measured.

Nuclei were found in all that were examined. These are usually sand grains, but one or two of coal were found. The number of concentric rings is usually two, but under higher magnification, four to nine laminæ may be seen. The outer zone is somewhat yellowish, probably stained from without; the inner zone is apparently more crystalline. Some oolites, in thin section, showed a rather fine-grained ground mass with a few scattered crystals. No trace of radiate structure was seen.

CHEMICAL COMPOSITION AND ORIGIN

The origin of the oolites is readily seen. They are chemical precipitates from the spray from the water, due probably chiefly to loss of carbon

TABLE I. *Dimensions of the oolites.*

Number measured	Diameter in mm.	Number measured	Diameter in mm.
1	0.7	14	1.9
0	0.8	9	2.0
3	0.9	3	2.1
5	1.0	7	2.3
8	1.1	1	2.4
9	1.2	3	2.5
12	1.3	1	2.6
24	1.4	1	2.7
18	1.5	0	2.8
7	1.6	4	2.9
13	1.7	0	3.0
11	1.8	0	3.1

dioxide; but possibly, to a slight extent, from evaporation. The chemical composition of the oolites and that of the solids in solution in the water used for spray is as follows:

TABLE II. *Analysis of Seymour City Water.*

	Parts per million		Parts per million
Silica	28.20	Probable combinations	
Iron and aluminum	7.20	Silica	28.20
Magnesium	33.42	Iron oxide and alumina	7.20
Calcium	60.15	Magnesium bicarbonate	201.30
Sodium	106.95	Calcium bicarbonate	243.00
Bi-carbonate	408.70	Sodium bicarbonate	80.65
Chloride	80.00	Sodium sulphate	102.24
Sulphate	69.13	Sodium chloride	137.61
Total			800.0

Analysis by Chemical Engineering Coporation, November, 1929, furnished by H. K. Jackson, City Secretary.

TABLE III. *Qualitative analysis of the oolites, exclusive of the nuclei.*

Silicic acid	Some	Magnesium	Much
Aluminum	Some, more than trace	Sodium	Very small trace.
Iron	Practically none	Sulphate	Trace
Carbonate	Much	Chloride	Trace
Calcium	Much		

Analysis made by the writer.

The presence of the silicic acid was somewhat surprising. The practical absence of sodium, chloride, and sulphate indicates that evaporation played a very minor part in the formation of the oolites. The water does not stand long around the base of the tower as the soil is porous and the water soon sinks away. There is sufficient movement to produce rotation of the nuclei and the oolites.

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A MINERAL ANALYSIS OF SOME COMMON SANDS AND THEIR SILICA CONTENT[1]

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ABSTRACT

The silica contents of nine common sands calculated from mineralogic analyses are compared with the silica contents determined by chemical analyses. The maximum difference, a deficiency of 2.46 per cent in the mineralogic analyses was for a sand containing 89.8 per cent silica by chemical analysis. The differences decrease with increasing silica content, although not in a very regular way. The maximum silica content by chemical analysis, namely, 96.03 per cent, differs by 0.78 per cent from the corresponding silica content in the mineralogic analysis.

A short method of mineralogic analysis gives results that differ by a minimum of 0.39 per cent and a maximum of 2.50 per cent from the results obtained by a detailed mineralogic analysis.

The detailed mineralogic analyses show: (1) The percentage of silica in the heavy mineral portion is fairly regularly inversely proportional to the percentage of heavy minerals in the sands. (2) The abundance of grains of the different minerals in the various grain sizes varies regularly with some minerals and irregularly with others. Some minerals, e.g., zircon, are markedly confined to certain sizes. (3) Minerals with good cleavage are not uniformly more abundant in the smaller grain sizes. Kyanite, for example, was found to be regularly more abundant in the larger grain sizes.

INTRODUCTION

The silica contents of nine common sands calculated from mineralogical analyses are compared with the silica contents determined by chemical analyses. The sands are commercial building, concrete, and plastering sands. They were obtained from tide-water localities of the Patuxent, Potomac, Patapsco, and Delaware Rivers. They were washed free of clay at the place of origin by the operators, and both the chemical and mineralogical analyses were made on this basis.

[1] The author is indebted to professor J. T. Singewald, Jr., for making available the chemical analyses, the samples of sand, and for advice in the investigation.

METHOD OF ANALYSIS

The following method of mineralogical analysis was used. The sands were first sieved into six grain sizes, namely, greater than 0.589 mm. (>28 mesh), 0.589 to 0.295 mm. (28 to 48 mesh), 0.295 to 0.147 mm. (48 to 100 mesh), 0.147 to 0.088 mm. (100 to 170 mesh), 0.088 to 0.062 mm. (170 to 230 mesh), and less than 0.062 mm. (<230 mesh). Fractions of the five sizes from greater than 0.589 to 0.062 mm., were separated into light and heavy portions by means of bromoform of 2.84 specific gravity. To remove thoroughly all iron staining, the light and heavy portions were boiled for five minutes in 1:1 HCl. The residues, excepting

those of the size larger than 0.589 mm., were washed and mounted in Canada balsam. A record of the weight of all portions was kept.

In the microscopic study, with rare exceptions, at least 100 grains of each mount were counted. By means of a mechanical stage successive rows of the grains were passed in a straight line across the field of vision, care being taken not to repeat the counting of any grain.

From these data, the distribution of the minerals in all four grain sizes between 0.589 and 0.062 mm., was calculated on the basis of volume percentage. These percentages were then converted into weight percentages by allowing for the specific gravity of the minerals. The latter furnished the starting point for calculating the total silica from a mineralogical analysis. In accounting for grains larger than 0.589 mm. and smaller than 0.062 mm., the same percentage distribution was assumed to obtain as in the sizes 0.589 to 0.295 mm. and 0.088 to 0.062 mm., respectively. Also, as no separations were made into light and heavy fractions of grains smaller than 0.062 mm., the same ratio of light to heavy was assumed to hold in this as in the 0.088 to 0.062 mm. size.

SOURCES OF ERROR IN THE CALCULATION OF THE PERCENTAGE OF SILICA

The assumptions just mentioned, together with the fact that loss of weight by boiling in acid was not determined, introduce some error into the calculations. Since the percentage distribution in the 0.589 to 0.295 mm. and the 0.295 to 0.147 mm. sizes are fairly alike in the light portions (see Table V), and the sum of the weights of all the light portions of each sample is in no case less than 90 per cent of the weight of the sample, little error is introduced by assuming a like distribution in the >0.589 mm. size.

Grains smaller than 0.062 mm. were found to make up at the most only 3 per cent by weight of the entire sample, so that little error is introduced in assuming a distribution in that size like that in the 0.088 to 0.062 size.

Only an inappreciable amount of calcium carbonate could have been lost by boiling in acid. The chemical analyses, Table I, show that the percentage of lime is quite low. The maximum values of 0.78 and 0.88 per cent are found in samples 6 and 7, but in these feldspars are abundant. There is, however, a greater probable error due to the destruction of the more basic feldspars and the loss of soluble iron in the acid treatment. In Table II the samples low in sericite show uniformly an excess of silica in the mineralogical analyses. Some lower silica minerals and minerals without silica apparently have gone into solution in those sands in the acid treatment leaving the remainder higher in silica than the original sand.

The samples high in sericite, on the other hand, show a deficiency of silica in the mineralogical analysis. This is explainable, in part at least, by the fact that what are classified

as sericite grains are mostly aggregates of quartz and sericite but were arbitrarily calculated as sericite.

To determine the loss of weight in all the light and heavy portions due to the acid treatment would have required considerable time. Therefore a representative portion of sample 3, with the greatest percentage of heavy by weight, namely, 8.5 per cent, one of sample 4, with the greatest percentage of silica in the chemical analyses, namely, 96.03 per cent, and one of sample 9, with the least percentage of silica in the chemical analyses, namely, 88.56 per cent, and the greatest percentage of sericite in mineralogic analysis, namely, 17.4 per cent, boiled in 1:1 HCl for five minutes and washed clean by decanting in the manner used for all samples. In the following table the percentage loss in weights on boiling in acid is compared with other data.

The $2\frac{1}{2}$ per cent loss of weight in sample 4 compared with the weight of the total heavy in that sample, that is, 2.6 per cent, shows that a good deal of that loss is attributable to the light portion. The $5\frac{1}{2}$ per cent loss of weight in sample 3 shows that an appreciable part of the heavy is soluble in HCl, and probably consists of soluble iron (see Table I). The $5\frac{1}{2}$ per cent loss could not have come mostly from the sericite scales due to the acid treatment and the subsequent loss of them in decanting, since sample 3 contains only 0.9 per cent more sericite than sample 4 yet the latter shows only a $2\frac{1}{2}$ per cent loss in weight. Even sample 9, which contains 17.5 per cent sericite, shows a loss of weight of only $4\frac{1}{2}$ per cent. The $4\frac{1}{2}$ per cent loss of weight in sample 9 is probably due to the floating off of sericite scales in decanting. This loss in weight should be evidenced in a higher silica percentage in the mineralogic than in the chemical analysis, as is true for samples 3 and 4; that it is lower is another indication that what are classified as sericite grains are aggregates of quartz and sericite.

That the silica in the mineralogic analyses for samples 3 and 4 is not higher than that in the chemical analyses by an amount equalling the percentage loss of weights in these samples due to boiling in acid, namely, $5\frac{1}{2}$ per cent and $2\frac{1}{2}$ per cent, is due in part to the floating off of the lower silica minerals in the decanting process, as sericite, for instance, and possibly to the solution of basic feldspars, and other lower silica minerals.

THE TABLES

Table I is a record of the chemical analyses and sources of the samples;

TABLE A. *Comparison of loss in weights on boiling in acid, in per cent.*

Sample	Loss in weight	Total heavy	Sericite by weight	Total silica by chemical analysis	Total silica by mineralogic analysis	Difference
3	$5\frac{1}{2}$	8.5	3.5	89.52	90.81	+1.29
4	$2\frac{1}{2}$	2.6	2.6	96.03	96.81	+0.78
9	$4\frac{1}{2}$	2.0	17.5	88.56	87.44	−1.12

Table II is a comparison of the amount of silica determined by chemical and mineralogical analyses; Table III gives a record of the weight percentages of light to heavy in each grain size and the percentage of the whole sample that is included in each grain size; Table IV shows the distribution of heavy minerals by volume percentage; Table V is a like record for the light minerals. Table VI records the relation between the percentage of silica by weight in the heavy portions to the percentage of heavy of the whole sample.

CONCLUSIONS

In a mineralogic study of nine washed recent river sands from the Middle Atlantic Coastal Plain, col-

TABLE I. *Chemical analyses of the samples, dry basis, in per cent.*

Sample	1	2	3	4
Silica	89.83	91.75	89.52	96.03
Alumina	4.21	3.02	4.71	0.91
Ferric Oxide	2.28	2.76	5.72	1.40
Titanic Oxide	0.28	0.35	1.09	0.08
Lime	0.27	0.20	0.10	0.04
Magnesia	0.15	0.11	0.06	0.01
Alkalies and loss on ignition	2.98	1.81	0.80	1.53
Total	100.00	100.00	102.00	100.00

TABLE I (*Continued*)

Sample	5	6	7	8	9
Silica	95.02	88.69	92.57	91.65	88.56
Alumina	1.94	5.43	3.86	4.19	5.21
Ferric Oxide	1.40	2.12	1.40	1.84	2.72
Titanic Oxide	0.15	0.48	0.23	0.20	0.28
Lime	0.07	0.78	0.88	0.10	0.15
Magnesia	0.03	0.14	0.05	0.08	0.13
Loss on ignition and alkalies	1.39	2.36	1.01	1.96	2.95
Total	100.00	100.00	100.00	100.02	100.00

Sample 1, Columbia Sand and Gravel Co., Washington, Building sand.
Sample 2, Columbia Sand and Gravel Co., Washington, Concrete sand.
Sample 3, Massaponax Sand and Gravel Co., Patuxent River, Washington Market, Building sand.
Sample 4, Massaponax Sand and Gravel Co., Patuxent River, Washington Market, Concrete sand.
Sample 5, Arundel Corporation, Patapsco Plant, Baltimore, Concrete sand.
Sample 6, Arundel Corporation, Spring Garden, Baltimore, Building sand.
Sample 7, Arundel Corporation, Spring Gardens, Baltimore, Concrete sand.
Sample 8, Charles Warner Co., Delaware River, Philadelphia Market, Plastering sand.
Sample 9, Charles Warner Co., Morrisville, Philadelphia Market, Concrete sand.

TABLE II. *Comparison of chemical and mineralogical analyses, in per cent.*

Sample	Silica by chemical analysis	Calculated silica	Difference	Per cent sericite by vol.	Silica calculated on basis of 0.295 to 0.147 mm., light, plus total silica from heavy	Difference	Silica calculated on basis of 0.589 to 0.295 mm., light, plus total silica from heavy	Difference
1	89.83	87.37	−2.46	10.6	87.33	−2.50	87.58	−2.25
2	91.75	91.72	−0.03	4.6	90.95	−0.80	91.99	+0.27
3	89.52	90.81	+1.29	2.9	90.47	+0.95	90.67	+1.15
4	.03	96.81	+0.78	2.1	95.64	−0.39	96.64	+0.61
5	95.02	95.90	+0.88	1.3	95.74	+0.72	95.99	+0.97
6	88.69	89.85	+1.16	3.1	90.07	+1.38	90.60	+1.94
7	92.57	94.00	+1.43	1.1	93.67	+1.10	94.34	+1.77
8	91.65	90.24	−1.41	11.6	89.92	−1.73	90.44	−1.21
9	88.56	87.44	−1.12	17.4	86.77	−1.79	87.54	−1.02

TABLE III. *Weight of light and heavy and of whole sample in each grain size, in per cent.*

Size	>0.589 mm.			0.589 to 0.295 mm.			0.295 to 0.147 mm.		
Sample	Total	Light	Heavy	Total	Light	Heavy	Total	Light	Heavy
1	49.8	97.1	2.9	36.1	96.0	4.0	11.5	89.0	11.0
2	31.6	98.7	1.3	56.7	97.8	2.2	10.5	89.0	11.0
3	22.8	94.0	6.0	55.1	96.2	3.8	17.9	81.2	18.8
4	61.2	97.7	2.3	27.9	97.8	2.2	9.5	95.6	4.4
5	66.7	97.5	2.5	26.6	98.5	1.5	6.1	96.7	3.3
6	1.8	97.7	2.3	25.4	97.7	2.3	50.6	95.8	4.2
7	74.4	98.9	1.1	16.8	97.6	2.4	7.4	94.6	5.4
8	28.8	99.3	0.7	53.2	99.2	0.8	17.1	97.9	2.1
9	53.7	97.5	2.5	38.1	98.9	1.1	7.5	97.3	2.7

TABLE III (*Continued*)

Size	0.147 to 0.088 mm.			0.088 to 0.062 mm.			<0.062 mm.	Total of entire sample	
Sample	Total	Light	Heavy	Total	Light	Heavy	Total	Light	Heavy
1	1.4	90.4	9.6	0.4	92.3	7.7	0.7	95.7	4.3
2	0.6	77.2	22.8	0.2	86.2	13.8	0.3	97.0	3.0
3	3.4	61.3	38.7	0.6	65.7	34.3	0.2	91.5	8.5
4	1.2	89.1	10.9	0.3	90.3	9.7	0.3	97.4	2.6
5	0.4	95.8	4.2	0.1	96.4	3.6	0.05	97.7	2.3
6	15.3	90.2	9.8	4.0	88.1	11.9	2.9	95.0	5.0
7	0.3	92.0	8.0	0.4	97.5	2.5	0.6	98.3	1.7
8	0.7	93.8	6.2	0.06	91.3	8.7	0.04	99.0	1.0
9	0.4	95.4	4.6	0.08	97.9	2.1	0.1	98.0	2.0

TABLE IV. *Distribution of heavy minerals by volume, in per cent.*

	Leucoxene				Other opaques				Hornblende			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample												
1	12.2	14.5	21.2	16.2	12.2	14.5	21.2	16.2	10.3	11.7	3.7	4.1
2	15.5	21.0	16.0	11.0	15.5	31.0	36.5	26.0	7.0	5.0	7.0	8.0
3	4.3	11.8	13.0	8.7	4.7	47.2	73.3	49.0	11.8	10.1	4.1	12.4
4	5.8	6.2	11.3	9.5	3.9	6.2	26.2	33.6	11.7	55.6	20.5	21.2
5	8.0	19.0	25.8	16.6	5.3	26.3	21.1	11.0	25.2	35.0	32.5	40.9
6	10.9	3.4	21.1	23.7	7.3	3.4	25.9	28.9	52.3	57.0	30.5	27.7
7	4.3	6.8	3.3	3.7	3.5	16.0	13.0	8.5	45.7	39.5	49.3	55.0
8	5.0	10.6	16.7	13.9	4.0	9.6	38.9	32.3	33.7	42.7	21.3	27.3
9	12.8	19.9	16.7	23.2	8.5	16.2	25.1	15.5	14.7	13.2	21.7	7.0

TABLE IV (*Continued*)

	Sericite > 2.84 sp. g.				Epidote				Garnet			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample												
1	47.4	30.2	13.2	10.0	7.7	16.5	25.8	31.5	1.3	0.3	0.3	0.5
2	23.0	10.0	6.5	2.0	24.0	23.0	15.0	33.0	3.0	1.0	0.5	2.0
3	14.7	4.6	1.5	3.1	1.0	3.1	3.9	18.9	6.6	2.4	0.8	0.9
4	29.7	5.1	12.1	5.6	2.1	11.3	13.0	19.1	3.4	1.4	0.6	0.5
5	25.2	1.8	2.1	1.8	5.9	8.4	7.7	11.5	4.4	—	0.3	0.3
6	10.2	4.9	3.6	3.3	4.2	9.6	2.6	3.6	0.7	1.0	2.6	1.5
7	—	2.5	—	—	7.1	15.3	20.4	22.2	2.1	2.1	—	0.5
8	34.8	21.4	1.8	4.4	10.1	9.8	5.1	5.2	—	—	3.2	1.2
9	36.0	27.9	9.1	3.5	8.1	3.9	4.0	6.8	—	2.6	1.1	1.0

TABLE IV (*Continued*)

	Hypersthene-Enstatite				Kyanite				Zircon			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.587 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample												
1	3.8	7.0	5.4	2.3	—	—	—	—	—	0.3	2.7	11.9
2	10.0	4.0	4.0	1.0	—	—	—	1.0	—	1.0	5.5	7.0
3	1.0	—	—	—	10.0	1.9	0.2	—	—	1.7	1.1	1.3
4	—	0.3	—	—	7.6	2.1	1.4	0.8	—	0.3	0.9	2.0
5	1.5	2.6	3.5	—	5.9	—	0.3	0.5	—	1.0	1.9	5.8
6	3.9	1.0	2.8	—	1.4	1.5	0.2	0.3	—	0.2	3.2	2.4
7	1.4	1.1	1.4	—	9.9	1.4	1.4	—	—	—	1.1	—
8	1.1	2.1	0.4	0.2	—	—	—	0.5	1.1	0.9	6.5	6.9
9	14.0	8.5	10.8	?	—	—	0.5	0.3	—	1.5	6.3	32.6

TABLE IV (*Continued*)

	Staurolite				Tourmaline				Rutile			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	.0888 to 0.062
Sample												
1	—	—	0.3	0.5	1.3	0.3	—	1.4	3.8	0.9	2.4	2.3
2	—	—	—	—	1.0	1.0	1.5	2.0	—	—	1.0	1.0
3	37.0	11.8	0.8	1.0	2.4	4.3	0.4	1.2	—	0.7	0.2	1.2
4	15.2	6.1	5.2	1.3	0.7	—	2.3	2.8	—	0.7	3.2	2.0
5	—	0.5	0.3	—	—	1.3	2.1	1.8	—	0.8	—	2.6
6	5.3	4.6	2.6	0.6	0.4	1.5	1.0	2.1	0.7	1.2	1.2	2.7
7	12.0	5.3	2.7	1.6	1.4	1.8	1.1	1.1	—	1.4	0.3	1.6
8	—	0.4	1.4	1.0	3.4	0.9	2.1	0.7	1.1	0.4	2.5	1.4
9	—	0.9	0.3	2.0	—	2.3	1.5	0.5	—	—	1.1	5.0

TABLE IV (*Concluded*)

	Muscovite				Tremolite			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample								
1	—	2.8	0.3	—	—	0.3	3.0	3.2
2	1.0	—	1.5	—	—	2.0	4.0	2.0
3	4.7	0.7	0.8	1.3	—	—	—	—
4	14.5	1.0	1.2	0.8	2.8	3.1	2.0	—
5	17.0	1.6	0.8	0.5	—	0.6	—	2.1
6	—	0.2	0.6	0.9	—	9.1	—	1.2
7	9.1	1.8	—	—	1.4	1.4	3.5	4.8
8	—	—	0.5	—	2.2	0.9	0.4	1.5
9	—	—	0.8	0.3	—	—	—	0.5

Chlorite, biotite, andalusite, titanite, anatase, zoisite, diopside, augite, diallage and actinolite are too sporadic in occurrence to be included in the table. Titanite and diopside, however, are not scarce in samples 5 and 9, respectively.

TABLE V. *Distribution of light minerals by volume, in per cent.*

	Quartz				Sericite <2.84 sp. g.				Orthoclase—Microcline			
Size in mm.	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.195 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample												
1	79	79	68	74	10	13	21	16	9	8	9	7
2	84	84	68	74	4	7	17	16[a]	10	6	6	6[a]
3	95	94	94	92	3	3	3	5	—	1	2	1
4	96	94	89	87	2	2	4	4	2	2	2	4
5	93	93	92	92	1	2	1	2	5	4	7	3
6	85	85	85	76	3	4	2	3	6	4	4	4
7	87	86	84	82	2	2	2	2	5	3	6	5
8	81	80	82	77	11	12	10	13	8	8	8	10
9	78	76	80	78	17	17	11	10	5	5	8	12

[a] Part of the sericite in the smaller sizes is clearly due to the alteration of feldspars.

TABLE V (*Continued*)

Size in mm.	Oligoclase				Andesine			
	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample			1	3				
1	—	—			—	—	—	—
2	1	1	1	1	—	1	—	1
3	—	—	—	1	—	—	—	—
4	—	1	1	2	—	—	1	—
5	1	1	—	2	—	—	—	—
6	1	2	2	4	1	1	2	2
7	3	4	4	4	—	1	—	1
8	—	—	—	—	—	—	—	—
9	—	1	—	—	—	—	—	—

TABLE V (*Concluded*)

Size in mm.	Albite				Muscovite			
	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062	0.589 to 0.295	0.295 to 0.147	0.147 to 0.088	0.088 to 0.062
Sample								
1	—	—	—	—	2	—	1	—
2	—	1	2	—	—	—	4	2
3	—	—	—	1	2	2	1	—
4	—	—	—	2	—	1	3	1
5	—	—	—	—	—	—	—	1
6	2	2	2	2	1	1	1	5
7	3	4	4	6	—	—	—	—
8	—	—	—	—	—	—	—	—
9	—	1	1	—	—	—	—	—

Sample 2, size 0.147 to 0.088 mm., Leverrierite, 2 per cent.
Sample 6, sizes 0.589 to 0.147 mm., 0.088 to 0.062 mm., Chlorite 1 to 2 per cent; 0.147 to 0.062 mm., Hydromicas 2 per cent.

TABLE VI. *Silica in the heavy portions.*

Sample	8	7	9	5	4	2	1	6	3
Per cent heavy in total sample	1.0	1.7	2.0	2.3	2.6	3.0	4.3	5.0	8.5
Per cent SiO_2 per one per cent heavy	0.397	0.351	0.358	0.377	0.365	0.278	0.323	0.294	0.234

TABLE VII. *Feldspars, in per cent.*

Size	28 to 48 mesh	48 to 100 mesh	100 to 170 mesh	170 to 230 mesh
Sample				
1	9	8	10	10
2	11	9	9	8
3	0	1	2	3
4	2	3	4	8
5	6	5	7	5
6	10	9	10	12
7	11	12	14	16
8	8	8	8	10
9	5	7	9	12

lected from several localities from the Delaware River to the Potomac River, the following results were found:

I. The Calculation of the Silica Percentage.

(1) The silica content of the sands calculated from their mineralogic analyses, taking into account all the mesh sizes, departed from chemical analyses of the same sands by amounts ranging from +1.43 to −2.46 per cent. These mineralogic analyses involved long, tedious operations and calculations.

(2) In the case of these particular sands, it was found that if the labor of the mineralogic analyses be greatly reduced by screening out at the beginning only the 0.295 to 0.147 mm. (48 to 100 mesh) size and calculating the silica content of that size only, that the calculated percentage will depart but little more from the chemical analysis than when all the mesh sizes are included in the calculations. The departures in this case are from +1.38 per cent to −2.50 per cent. Or, in like manner, only the 0.589 to 0.295 mm. (28 to 48 mesh) size may be considered, and the calculated silica content departs from the chemical determination from +1.94 to −2.25 per cent. The lesser extreme negative departures involved in calculating the total silica from the 28 to 48 mesh size indicate that this size is preferable to the 48 to 100 mesh size for the silica calculations. Furthermore, the calculations based on the 28 to 48 mesh size give positive departures, that is, the silica in the mineralogic analyses is greater than that in the chemical analyses (see Table II), except for samples 1, 8, and 9, which contain rather large amounts of sericite. These positive departures for the low sericite samples are what would be expected from a consideration of the data in Table A. The previous discussion of that table is not repeated here.

(3) Since the ratio of the percentage of silica by weight in the heavy portions to the percentage of heavy in the whole sample decreases regularly with an increasing amount of heavy (see Table VI), little error would be introduced by calculating the silica in the total heavy portion on the basis of Table VI.

(4) A short method, then, of determining the silica in the sands involves the following operations: (*a*) Separating a representative portion of the whole sample into light and heavy parts, (*b*) Weighing the light and heavy parts and calculating the percentages of each, (*c*) Sieving out the 0.589 to 0.295 mm. (28 to 48 mesh) or the 0.295 to 0.145 mm. (48 to 100 mesh) grain size, boiling in 1:1 HCl acid for 5 minutes, washing, decanting, drying, and mounting in Canada balsam, (*d*) Counting and identifying 100 successive grains, (*e*) Calculating the total silica in the light portion by assuming the same distribution of minerals in all the light mesh sizes as in the 28 to 48 or the 48 to 100 mesh sizes. The volume distribution of the minerals in the mineralogic analysis may be assumed to be the same as the weight distribution, since the specific gravities of quartz and the feldspars are not enough different to affect appreciably the result. In the case of the higher sericite sands, such an assumption would reduce the magnitude of the negative departures of the silica in the mineralogic analyses from the silica in the chemical analyses. (*f*) Adding to this amount the total silica in the heavy calculated by using Table VI.

(5) The calculations based on the 28 to 48 mesh size give, for the samples containing less than 3 per cent sericite, an excess of silica over that in the chemical analyses of about 1 to 2 per cent. Calculations based on the 48 to 100 mesh size give, for like samples, an excess of silica of about −one-half to 1½ per cent. Those samples carrying a large amount of sericite, 10 to 17 per cent, show a deficiency in the silica of the mineralogic analysis, ranging from −1.21 to −2.25 per cent for calculations based on the 28 to 48 mesh size, and a deficiency of −1.73 to −2.50 per cent for calculations based on the 48 to 100 mesh size. As explained previously, a large percentage of the grains classified as sericite are aggregates of quartz and sericite, and the deficiency of silica in the mineralogic analyses is probably due to this arbitrary method of classification.

II. Summary of the Distribution of the Minerals.

(1) The feldspars are not uniformly, though usually, more abundant in the smaller grain sizes (see Table V). In sample 2 they are more abundant in the 28 to 48 mesh size than in any smaller size. The distribution may be tabulated as follows:

(2) Sericite, <2.89 Sp.G., may be more abundant in either the larger or smaller sizes (see Table V).

(3) Quartz is more abundant in the coarser sizes, except in sample 9, in which the 170 to 230 mesh size contains as much quartz as the 28 to 48 mesh size (see Table V). The excess percentage of quartz in the 28 to 48 mesh size over that in the 170 to 230 mesh size ranges from 0 to 10 per cent.

(4) Sericite, >2.84 Sp.G., and muscovite >2.84 Sp.G., decreases regularly in abundance from the 28 to 48 mesh size to the 170 to 230

mesh size (see Table IV). These sericite grains are practically free of quartz.

(5) The distribution of epidote is irregular (see Table IV).

(6) Garnet tends to be abundant in the coarser sizes.

(7) The abundance of kyanite and staurolite in the 28 to 48 mesh size and their rapidly decreasing occurrence in the smaller sizes is marked.

(8) The regular and rapid increase of zircon in the smaller sizes and its almost complete absence in the 28 to 48 mesh size is very pronounced.

III. Especially Notable Features in the Distribution of the Minerals.

A cursory search of the literature yielded corroborative evidence that zircon occurs in the smaller mesh sizes. The occurrence of detrital zircon in the 28 to 48 mesh size, except in minute amounts, is probably quite rare.

The abundance in the larger grain sizes of minerals with well developed platy cleavage, such as the feldspars, and especially kyanite, was unexpected. The common opinion is that minerals with platy cleavage are likely to be more abundant in the smaller mesh sizes, due to easy breaking along cleavage planes. This is certainly not true, however, of the kyanite in these sands.

NOTES

THE ORIGIN AND LIMITS OF A ZONE OF ROUNDED QUARTZ SAND OFF THE SOUTHERN NEW ENGLAND COAST

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Dr. Alexander, in a recent article[1] on the petrography of some sediments from the continental shelf, discusses the origin of a zone of rounded quartz sand which was encountered off the coast of southern New England. Since the first dredging trips were made by the Woods Hole Oceanographic Institution new data have been accumulating which lend further weight to a somewhat different interpretation.

In the first place, the zone is of much greater extent than was at first supposed. It is picked up 15 miles off Marthas Vineyard in 144 feet of water, about the same distance off the eastern end of Long Island in 184 feet, and 20 miles off Block Island in 194 feet. On the two easternmost lines this rounded sand is found over the edge of the shelf and on the Vineyard traverse to a depth of 1773 feet which is the limit of dredging. The rounded grains have a fairly definite size range, running from the 48 mesh, or 0.295 mm. sieve, to the 14 mesh or 1.165 mm. Every sample contains some grains with a mat surface, and the coarse fraction of the samples from the outer parts of the shelf consist almost entirely of such grains within the limits given above. Certain samples from the middle section of the shelf consist largely of bright grains, although the high degree of rounding continues. It so happens that the samples with these clear grains also contain the greatest amount of silt and clay, running as high as 80 per cent. Numerous holothurians and worms of various kinds are abundant in this region. It is well known that these animals are mud-feeders, and therefore the original ground-glass surface may possibly have been removed by solution and polishing by repeated passages through the intestines.

I originally advanced the opinion that this sand, because of its characteristics, must be associated with wind action.[2] The high degree of rounding is indicative of long history. There is nothing approaching it in the modern dunes of Cape Cod. The great numbers of wind-cut stones on the Cape, and outlying islands, give evidence of the vigor of this wind during the Pleistocene. Its present position offshore can be adequately explained by the fluctuations of sea-level which must have occurred during and after this period.

Dr. Alexander proposes the theory that this material was produced on the present-day beaches of Cape Cod, and transported to its position by now existing currents. It is doubtful if a sand equalling the St. Peter sandstone in degree of rounding could be produced exclusively by wave action. We have, however, some positive evidence at hand. Mr. Marshall Schalk, in the course of his shoreline studies at the Woods Hole Oceanographic Institution, collected over 150 samples from these same outer beaches of Cape Cod, from Long Point to Monomoy, a distance of about 50 miles. Only two or three grains of

[1] *Jour. Sed. Petrology*, vol. 4, pp. 12–22, 1934.

[2] Stetson, H. C., Sediments of the Continental Shelf off the Northeastern United States: (Abstract) *Geol. Soc. America, Bull.*, vol. 44 p. 206, 1933.

rounded, frosted sand were found in the entire suite. A little of this material is to be expected, as some must be present in the cliffs in the neighborhood of the wind-cut stones. What the total amount is, cannot be determined, but we do know that its presence is, to all intents and purposes, completely masked by the angular grains which make up the present beaches. The same is true of the south beach of Marthas Vineyard, from which Mr. Schalk took 40 samples.

As for transportation, there is, it is true, a general surface drift around Cape Cod to the westward. Even if this current extends to the bottom, which as yet has not been demonstrated, its velocity would have to be greatly increased over that of the surface in order to move sand of the size which we find here. Finally, much of this sand is mixed with silt and clay, and in the offshore regions where the coarser grades consist entirely of rounded grains, the silt-clay fraction makes up from 20 to 80 per cent of the sample. It is obvious that two environments are represented here. A current powerful enough to sweep in the sand would at the same time carry off the silt and clay.

LUCKE, JOHN B., *A Study of Barnegat Inlet, New Jersey, and Related Shoreline Phenomena*, Shore and Beach Journal of the American Shore and Beach Preservation Association, vol. 2, no. 2, 1934.

The area described in this study is taken as a typical example of a tidal inlet on a shoreline of emergence. The relief of the area does not exceed 25 feet. Barnegat Inlet is one of the series of inlets that cut the offshore bar which lies along the New Jersey Coast south of Point Pleasant. The purpose of the study was to establish the origin and sedimentary nature of the tidal delta associated with the inlet and to determine the significance of tidal deltas in the history of a shoreline of emergence.

The United States Coast and Geodetic Survey has mapped the position of Barnegat Inlet six times since the first accurate charting in 1839. These charts show a progressive southward migration of the inlet over a distance of about a mile. It is assumed that if the delta associated with the inlet had remained stationary, the sediments would decrease in average size not only out into the lagoon towards the mainland, but also north and south from the inlet. The mechanical composition of the sediments corroborates the charts of the inlet showing a southward migration, the coarser sediments being obtained from those areas over which the inlet has migrated. The mechanical composition of the sediments does not indicate that the position of the inlet was ever far north of the position shown in the chart of 1839, nor south of the present position. The minerals of the sandy sediments that comprise the delta are, in general, similar in kind to those found in the New Jersey beach sands. The delta deposits are unstratified, a condition, according to Lucke, probably resulting from the uniform character of the material. The abundant life of the area is that typical of shoal marine waters. In spite of the abundance of forms now living in the area, the delta shows very little in the way of fossils of macroscopic size. The author offers no explanations to account for this rarity of fossils. The shallow water nature of the sediments is reflected in the surface markings. Localized areas of black organic mud are noted on the tidal delta, but such are not encountered in the borings.

Lucke finds that the inlets of the stationary type produce quite a different pattern of shoal and marsh fillings in the lagoons than do inlets of a rapidly migrant type. By means of hypothetical cases he deduces the sequences of such fillings and checks these successfully against charts of the New Jersey coast.

The paper is very well illustrated with many photographs and drawings. The airplane views, would be more valuable if described in the titles.

The deductive method is followed throughout.

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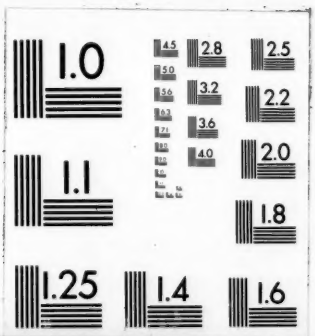
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